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RELATIVE TOXICITY TESTING OF
SPACECRAFT MATERIALS

II. AIRCRAFT MATERIALS

ANNUAL REPORT TO
NASA JOHNSON SPACE CENTER
HOUSTON, TEXAS 77028



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Date of Report November 6, 1980
Report Prepared by W.H. Lawrence, Ph.D.
Associate Director and
Head, Animal Toxicology Section

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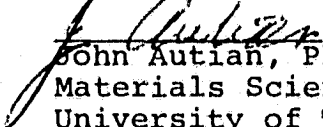
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Introduction

There have been a number of aircraft accidents involving fires in which loss of life occurred. Subsequent investigations indicated the extent of damage and heat from the fire should not have prevented safe escape of the occupants in some of these instances. It was therefore presumed that the victims had been overcome by toxic gases generated by the relatively minor fire. Such unfortunate incidents, however, have served as a stimulus for the search for new or alternative materials which pose a smaller risk to aircraft occupants.

Two basic approaches, as well as a combination of both, have been employed to address this problem: (a) reduce flammability of the materials by incorporation of flame retardant chemicals in the materials, or (b) selection of materials which produce the least toxic pyrolysis/combustion products during thermodegradation. The former method may result in a material which is difficult to ignite and which will not support combustion. However, if sufficient heat is generated from another source, these materials will decompose and the pyrolysis/combustion products are often more toxic than those produced by the same materials without the flame retardant added (1, 2). Since there is usually two or more materials which possess

¹W.H. Lawrence, "Toxicity of Pyrolysis Products: Influence of Experimental Conditions, and Presence of a Flame Retardant". Presented at the Flame Retardant Chemicals Association Fourth Annual Meeting, March 20-23, 1977, Washington, D.C. and published in Association's Collection of Papers (1977).

²L.J. Nunez, G.W. Hung and J. Autian, "Toxicity of Fabric Combustion Products", Journal of Combustion Toxicology, 3:371-380 (1976).

satisfactory properties for each application, the second approach is to evaluate these materials for thermodegradation characteristics and toxicity of products produced, and then to select the material which poses the least toxic hazard to the occupants from a relatively small fire in the aircraft.

Purpose of Work. The primary objective of this work was to obtain information about the relative toxicity of thermodegradation (pyrolysis/combustion) products of aircraft materials supplied to us by the Technical Monitor. Two approaches were taken to assess the biological activity of the pyrolysis/combustion products of these materials: (a) determine the acute lethality to rats from inhalation of these pyrolysates, and (b) examine the tendency for sublethal exposures to the pyrolysates to disrupt behavioral [shock-avoidance] performance of exposed rats. The relative importance of lethality vs. behavioral effects in selection of a material may be dictated (or influenced) by whether or not individuals potentially exposed to such products, would have an opportunity to escape if they were behaviorally capable of doing so. If so, the second parameter would assume greater importance, but if not, the first parameter may be of much greater importance in selecting materials.

Materials and Methodology

Test Samples. Five (5) aircraft materials were included in this study. The code designation and description, as provided by the Technical Monitor, of each test sample are

presented in Table 1.

Method of Sample Pyrolysis/Combustion. All samples were pyrolyzed/combusted directly in the rat exposure chamber using an electric furnace, and all products mixed thoroughly with the chamber atmosphere by an electric fan located inside the exposure chamber. An experimental constraint which influenced pyrolysis/combustion of samples was that of chamber temperature, i.e., the chamber temperature was not to exceed 35°C (95°F). To accomplish this, the sample was pyrolyzed rapidly (~10 minutes) and the furnace removed from the chamber to reduce added heating of the chamber atmosphere from the furnace as it was cooling.

The first part of this study was performed using a conical ceramic furnace with a platinum wire heating element to provide rapid, intense heat for thermodegradation of the test sample. Initially this system worked quite well, but after awhile the furnace began to fail, often breaking down in the middle of an experiment. It was found that the exposed platinum wire was apparently reacting with the pyrolysis products, which resulted in its failure as a heating element. Thus, this procedure permitted rapid attainment of a high temperature (well in excess of 1,000°C) to produce rapid thermodegradation (pyrolysis) of the test sample, and thereby minimizing heating of the exposure chamber atmosphere. On the other hand, the platinum heating element degenerated with use, and it was also difficult to quantitatively recover sample residues thereby providing, at best, only estimates for completeness of degradation.

A new furnace was built which was used in the rest of

the study. It contained heating elements embedded in a high temperature ceramic type material in the bottom and four sides of a rectangular chamber 3" x 3" x 5". These heating elements had a maximum temperature rating of 1,200°C., thus exceeding the 1,000°C capability which we desired. A removable stainless steel rectangular cup, with internal dimensions of 2.5" x 2.5" x 4.5", was constructed to fit closely inside the space formed by the heating elements. Since this furnace requires much longer to pyrolyze a sample starting at room temperature, than the platinum-wire one, pyrolysis was accomplished by pre-heating the furnace (outside of the chamber) to about 800-900°C, then placing the stainless steel cup (containing the test sample) in the furnace opening, and immediately placing the furnace and sample in position to pyrolyze the sample directly in the rat exposure chamber. When pyrolysis was completed, this furnace, like the other one, was removed from the exposure chamber to prevent additional heating of the chamber atmosphere by radiation from the furnace. The time required for thermodegradation, and the increase in chamber temperature, varied depending upon sample and quantity of sample. In most cases, however, this could be accomplished and still maintain the chamber temperature less than 35°C (95°F). One advantage to this system was that it provides for a more accurate and easier determination of sample residue.

Thermogravimetric Analyses (TGA). The thermodegradation characteristics of each sample were determined in air and nitrogen. This provided general information about the temperature

required to initiate degradation, to complete degradation, expected percent degradation, and some indication of the importance of oxidative processes for degradation.

LD₅₀ Determinations. The lethality of each sample, except one where there was an insufficient quantity of sample, was determined by pyrolyzing specific weights of sample and exposing a group of 4 male Sprague Dawley rats to the pyrolysates for 30 minutes after completion of pyrolysis of each sample weight. LD₅₀s were calculated for the samples based upon chamber deaths, deaths occurring within 48 hours, and those occurring within the 14-day post-exposure observation period. The chamber atmosphere was analyzed for selected gases by use of gas detector tubes or gas chromatography, or both. Carboxyhemoglobin (COHb) levels were determined in rats which died in the chamber. Animals were autopsied, when they died or were sacrificed after 14-days, and tissues from most of these preserved in buffered formalin and subjected to histopathologic evaluation. The actual LD₅₀s, expressed as initial weight of sample pyrolyzed to kill 50% of exposed rats, was calculated by Cornfield and Mantel's modification of Karber's method.⁽³⁾

Behavioral Studies. The subjects were adult male Sprague Dawley rats (about 275-325 gm), which were identified and housed individually in stainless steel cages. They had free access to fresh tap water and laboratory rat chow. The behavioral apparatus (shock chamber) was approximately

³J. Cornfield and N. Mantel, "Some new aspects of the application of maximum likelihood to the calculation of the dosage response curve", Journal of the American Statistical Association, 48:181-210 (1950).

7.5 x 9.5 x 7.75 inches, with a Gerbrand lever mounted in one end. The floor and two sides consisted of 1/16 diameter stainless steel rods, mounted about 0.5 inches apart, and connected to a shock source and shock scrambler. This shock chamber was placed inside the inhalation exposure chamber used in the LD₅₀ studies, which has a volume of approximately 200 liters.

The rats were trained on a Sidman avoidance schedule, with a shock-shock interval of 5 seconds, and a response-shock schedule interval of 20 seconds. The shock duration was 0.5 seconds, with an intensity of 1.3 milliamps. Thus, if the rat did not press the lever at all, he would receive 12 shocks/minute, but appropriate lever presses would permit complete avoidance of shocks. During training, each rat was placed in the apparatus for one hour per day. When the average number of shocks an animal received during the hour was 2.5/minute or less, and the response rate was stable for 2 consecutive days, he was considered for use as a test subject. This usually occurred in 2 weeks or less, and animals that did not reach this criterion after 2 weeks of training were usually rejected from the study.

After this training, the first stage of testing consisted of two 70 minute sessions held on two consecutive days, during which cumulative shock avoidance rates were monitored every 10 minutes. If, during the first session, a subject's rate of avoidance decreased so that he received an increased number over any 20 minute period, he was returned to the training

conditions until his performance was stabilized. He was then retested. The purpose of these sessions was to obtain a steady baseline measure against which the performance on the day of pyrolysis testing could be assessed.

On the day of pyrolysis testing, the rat is placed in the behavioral apparatus and a 30-minute control performance is obtained. If the rat's avoidance performance is adequate and reasonably stable during this time, this is used as the pre-burn control (or response for the pre-burn period) and the experiment is continued. However, if the rat's avoidance behavior is inadequate or very unstable, the sample is not burned, and the rat is returned to training or discarded. If this pre-burn control is satisfactory, the chamber door is secured and the designated sample is pyrolyzed/combusted in the chamber. The animal's shock-avoidance performance during pyrolysis/combustion is recorded, and performance is recorded at 5 minute intervals for the ensuing 30 minutes while the inhalation chamber remains closed.

The pre-burn control period was a minimum of 30 minutes, and frequently a little longer; the duration of pyrolysis/combustion varied from about 6 to 18 minutes, depending upon the sample. The maximum temperature in the chamber was found to range from 84°F to 93°F. In each instance, the rat's performance was monitored and recorded for at least 30 minutes after completion of pyrolysis/combustion.

As a basis for comparison of "equivalent" non-lethal exposures, one half (1/2) of the LD₅₀ sample weight was chosen

for the working level for 4 of the 5 samples (Y-7191, Y-7192, Y-7212, and Y-7214). The fifth sample (Y-7213), an estimated LD₅₀ was chosen from rather inadequate data, but due to the limited quantity of sample it was not possible to determine the LD₅₀ and also conduct behavioral tests. Each sample material was tested by exposing four trained rats to its pyrolysate. Assignment of a particular rat to a specific sample and order of testing was semi-random, being determined by availability of the sample, the trained rats available, and the needs for obtaining a block of 4 rats per sample. Each rat was exposed to a pyrolysate of only one sample (experimental run).

In addition, one group of 4 rats was exposed to the pyrolysate from one-fourth (1/4) of the LD₅₀ of Y-7192. This group was included in the statistical analyses along with the other five groups. Also, two of the other samples were tested at quantities other than one-half of the LD₅₀; however since there were fewer than 4 rats/group, these were not included in the statistical analyses, but the responses are included in the behavioral figures.

Results and Discussion

Thermogravimetric Analyses (TGA). A computer plot of the thermodegradation of each test material is presented in Figures 1 through 21. Each material was tested in air and nitrogen to obtain an indication of the importance of oxidative degradation vs. a non-oxidative atmosphere. The polyimide

(Y-7191 and Y-7214) exhibited less loss of weight during heating in nitrogen than in air, while the polyurethane (Y-7192) and 3M Silicone on Fiberglass (Y-7213) gave similar values both for air and nitrogen. Results from the composite material (Y-7212: Orcon Kapton with Kel-F and AB 312 Ceramic Filler and Nylon Fibers) were inconsistent between the two runs in air, which may have been due to non-uniform distribution of the sample tested; thus there is probably little or no difference in thermodegradation between air and nitrogen atmospheres.

During the TGA runs, it was noted that the platinum weighing pans tended to fail (develop a hole, etc.) after repeated use. This would suggest the possibility of reaction between the pyrolysis products and the platinum, an observation also noted with the platinum wire furnace. Therefore, the TGA data must be considered as approximations where the patterns of decomposition of samples are probably real, but too much significance should not be attached to exact information from sample residue weights or percentages.

Significant information from the TGA experiments are summarized and grouped by sample, and presented in Tables 2 through 6.

Comparative Toxicity of Samples. The LD₅₀ values for each sample material, for chamber deaths, cumulative deaths through 48 hours, and cumulative deaths through 14 days post-exposure, are presented in Table 7. As indicated in Table 7, there was not enough of sample Y-7213 (3M Silicone on

fiberglass), to complete the LD₅₀ determination and to also conduct the behavioral study, therefore only partial lethality data are available on this sample. Pyrolysis/combustion of 25 gm of this sample did not kill any rats (0% mortality) while a 45 gm sample size killed all of the exposed rats (100% mortality). Based upon these criteria, the polyimide foam sample (Y-7191) was the most toxic of these samples when subjected to pyrolysis/combustion in the exposure chamber, while, except for Y-7213 (3M Silicone on Fiberglass), the polyurethane foam (Y-7192) was the least toxic. [The pyrolysates from the polyurethane foam, however, exerted a marked effect upon trained behavior of exposed rats, *q.v.*]

Table 8 incorporates theoretical percent decomposition of samples (from TGA data) with the sample weights required to kill 50% of the exposed rats. Three of these samples (Y-7214, Y-7192, and Y-7191) should be essentially completely degraded during the pyrolysis/combustion procedure, while about three-fourths of one sample (Y-7212) and one-fourth of the other sample (Y-7213) would be expected to be pyrolyzed by these conditions. Adjusting the LD₅₀ values for expected extent of thermodegradation, however, does not change the ranking of these materials, on the basis of toxicity, from that obtained by initial weight of sample required to kill 50% of the exposed rats. Rankings of these samples based upon lethality, both from initial sample weights and from theoretical quantity pyrolyzed/combusted, are presented in Table 9.

Acute toxicity data from the pyrolysis/combustion products of these samples are shown in Tables 10 through 14. These summarize mortalities, COHb levels, and selected gas analyses tabulated against the initial sample weight pyrolyzed/combusted.

Gas detector tubes, although possibly exhibiting some degree of non-specificity in such a complex mixture of thermodegradation products, were used to screen the chamber atmosphere for chlorine, hydrogen sulfide, oxides of nitrogen (NO/NO_2), vinyl chloride, and acetone. In only a very few cases did the detector tube indicate more than 10 times the TLV concentration. Noteable, was the H_2S concentration of $>0.16\%$ (TLV = 10 ppm) for the polyimide sample, Y-7191, but not for the other one, Y-7214.

It is often difficult to select a "toxic" value for many of these for a 30-minute exposure. The TLV is supposed to be a "safe" human exposure level for 8 hours per day, 5 days a week. The TLV for HCN is 10 ppm ($11 \text{ mg}/\text{M}^3$) and CO is 50 ppm ($55 \text{ mg}/\text{M}^3$). Published data indicate the lowest known lethal concentration of HCN to humans is about 110 ppm ($120 \text{ mg}/\text{M}^3$) for a 1 hour exposure or about 185 ppm ($200 \text{ mg}/\text{M}^3$) for 10 minutes of exposure (4). For carbon monoxide, it has been reported that 1,000-1,200 ppm (0.10-0.12%) produces unpleasant but not dangerous symptoms in humans after 1 hour exposure; a level of 1,500-2,000 ppm (0.15-0.20%) is dangerous when exposed for 1 hour; and a level of 4,000 ppm (0.40%) or greater may be fatal in less than 1 hour (5).

Pathology. Animals which died in the chamber and representatives of those surviving the 14-day post-exposure observation period were autopsied; major organs were removed and fixed

⁴Y. Henderson, H.M. Haggard, M.C. Teague, A.L. Prince, and Wunderluch, Journal of Industrial Hygiene, 3:79 (1921), cited in Industrial Hygiene and Toxicology, Second Revised Edition, Volume II, D.W. Fasset and D.D. Irish, Editors, Interscience Publishers, New York (1963), p. 930.

⁵N. Irving Sax, Editor, "Dangerous Properties of Industrial Materials", 5th Edition, Van Nostrand Reinhold Co., New York (1979), p. 728.

in formaldehyde solution, processed, and examined histologically for evidence of tissue damage. Post-exposure (delayed) deaths were similarly treated if possible, i.e., unless autolysis rendered the procedure useless. Histopathologic evaluation utilized a three-step process. First, the primary comments of the pathologist were recorded from microscopic examination of the specimen; second, a summary of these were tabulated by weight of sample pyrolyzed and also indicating time of specimen relative to time of exposure; and third, an evaluation of these observations to indicate those effects (both acute and delayed) thought to have resulted from inhalation of pyrolysis/combustion products of each material tested. As an example, sample forms used for primary comments and tabulated summary for two of these materials are shown in Exhibit 1, at the end of this report. The third step, "Summary of Histopathologic Evaluation", as completed by the pathologist, is presented for each sample material in Tables 15-19.

It might be pointed out that material, probably pyrolysis debris, was noted in the lungs of some rats. Two of the four rats that died in the chamber from pyrolysis/combustion of 45 gm of Silicone on Fiberglass (Y-7213) were examined histopathologically, and both showed evidence of massive inhalation of pyrolysis debris. Thus death of these rats may have resulted from mechanical blockage of respiration, rather than from toxic gases.

Behavioral Studies. Figures 23 through 51 graphically depict the individual shock-avoidance behavior of 29 trained

rats when exposed to pyrolysis/combustion products of these five samples. Since the process of pyrolysis also increases the temperature in the exposure chamber, Figure 22 is included to show that heat alone (up to 95°F or 35°C), without any pyrolysis products, did not adversely affect the rat's shock avoidance activity.

Identification of terms used in discussing behavioral analyses

Group I Rats ...exposed to 1/2 of LD₅₀ of Y-7192 (Polyurethane Foam)

Group II Rats ...exposed to 1/4 of LD₅₀ of Y-7192 (Polyurethane Foam)

Group III Rats ...exposed to 1/2 of LD₅₀ of Y-7214 (Polyimide Foam, #720-1)

Group IV Rats ...exposed to 1/2 of LD₅₀ of Y-7212 (Orcon Kapton with Kel-F)

Group V Rats ...exposed to 1/2 of LD₅₀ of Y-7191 (Polyimide Foam)

Group VI Rats ...exposed to 1/2 of the estimated LD₅₀ of Y-7213 (3M Silicone on Fiberglass)

Day 1 ...first of two consecutive 70-minute control runs prior to testing of sample

Day 2 ...second of two consecutive 70-minute control runs prior to testing of sample

Day 3 ...the day in which the trained rat was exposed to pyrolysates of the test sample.

Time Intervals ...The first three 10-minute periods of Days 1 and 2 were conceptually compared to the three 10-minute periods of Day 3, i.e., the "pre-burn" control. The fourth 10-minute period of Days 1 and 2 relate to the pyrolysis time of Day 3. The fifth, sixth, and seventh 10-minute periods of Day 1 and 2 were used for comparison to the three 10-minute periods comprising the 30 minute "post-burn exposure" period of Day 3.

An analysis of variance for weighted means with factors of Groups (6 levels) [$1/2$ of LD_{50} for each sample plus $1/4$ of LD_{50} for Y-7192], Days (3 levels), and Time (7 levels) was performed. The time levels were defined as six 10 minute intervals and one interval of variable time, being the 4th 10 minute interval on the first two days of testing and the interval of pyrolysis from 6-18 minutes on the third day. Days were defined as each day of testing. Groups were defined as sets of four subjects each assigned to a separate sample being evaluated, or in the case of Group II, a different fraction of one of the samples.

Summary Table for the Analyses
of Variance

Source	Sum of Squares	Df*	Mean Square	F-Ratio	Prob.
Groups (A)	57.5509	5	11.5102		
Between Error	197.251	18	10.9584	1.050	0.419930
Days (B)	70.4548	2	35.2274	12.733	0.000175
AB	178.305	10	17.8305	6.445	0.000055
Within Error	99.5957	36	2.76660		
1					
Time (C)	20.7491	6	3.45818	4.738	0.000438
AC	69.9317	30	2.33106	3.194	0.000031
Within Error	78.8195	108	0.729810		
2					
BC	108.911	12	9.07590	14.023	0.000000
ABC	220.731	60	3.67885	5.685	0.000000
Within Error	139.799	216	0.647217		
3					
Total	1242.10	503	2.46938		

Group differences, collapsed over Days and Time, were non-significant. A significant difference was found over days of testing and a significant interaction between Groups and Days. A significant difference was found over Times and over Times

by Days. A Groups by Time interaction was significant as well as a Groups by Time by Days interaction.

Newman-Keuls post hoc tests for significance were computed.

Factoring Groups, there proved to be no significant difference between any two Groups on Day 1 or Day 2 of testing. On Day 3, Group I statistics proved to be significantly different from those of all other Groups; from Group V at the 0.01 level and from Groups II, III, IV and VI at the 0.05 level. No other comparison between Groups on Day 3 yielded significance. Factoring Days, Group I showed no difference in performance between Days 1 and 2, but showed a significant difference between Days 3 and 2 and Days 3 and 1 at the 0.01 level. The mean shocks per minute score for Day 1 was 1.74, Day 2 was 1.76, and Day 3 was 5.24. Further, comparing Day 3 with Days 2 and 1, Group 1 showed a significant difference at the 0.01 level during the burn period as well as the 3 subsequent time intervals, but their controls (Days 1 and 2) showed no difference in shocks per minute during these intervals.

Comparison of pre-burn and post-burn intervals for Group II is also of interest. Although no difference was found during pre-burn intervals or the 4th interval (during burning) between Days 1, 2 or 3, the average scores for the post-burn period shows a difference between Day 3 and Days 1 and 2 at the 0.05 level. The mean shocks per minute were as follows:

Day 1, 1.96; Day 2, 1.81; Day 3, 3.02.

Group III also showed a difference significant at the 0.05 level between Days 1 and 3 during the 5th time interval, the first post-burn interval, (Day 1, 1.63; Day 3, 2.50) but showed no difference when the post-burn scores were averaged.

Factoring Days, Group V showed a significant change from Day 1 to Day 3 at the 0.05 level, but this change was in the direction of improvement, the means being 2.09, 2.00, and 1.71 respectively. Further analysis showed this difference to be due to improved performance during the post-burn period of Day 3.

Except as discussed above, all other differences found to be significant over Days and over Times were in the direction of improvement with continued performance. The high level of significance found for Day and Times appears to be accounted for by a practice effect. In all cases there were no differences found to be significant between Days 1 and 2 or between Times 1, 2 and 3 that were accounted for by a decrement in performance. Increases in the number of shocks per minute, which were related to factors with statistically significant differences, appeared only during the burn and post-burn periods of Day 3.

Of the five samples tested, only sample Y-7192 (sample assigned to Group I) affected avoidance behavior to the extent of producing a significant increase in number of shocks received during the burn and post-burn periods, when Group I scores were compared with scores of the other groups. This effect

was seen in all subjects tested. During the burn period, rate of avoidance behavior decreased, and continued to do so throughout the test period with no incidence of return to pre-burn levels of avoidance. All animals spent some periods of the burn lying on the floor of the apparatus making no apparent response to the shock. The extent to which this phenomenon occurred was variable from subject to subject, but the pattern was similar.

Sample Y-7214 was assigned to Group III. Although average scores from the entire post-burn period for this group did not differ significantly from those of any other group with the exception of Group I, analysis of performance for each post-burn time interval, comparing the three days of testing for this group, showed that a significant change occurred from Day 1 of testing to Day 3 during the fifth time interval (the first 10-minute post-burn interval on the third day). This change was in the direction of an increase in number of shocks received (a decrease in % shock avoidance). In all other groups (with the exception of Group I) the tendency was for shock-avoidance to remain stable or to increase during this period. This time period appeared to be critical for Group III subjects, since for each subject the decreased rate of avoidance was followed by a return to levels close to those set in the pre-burn period. In half of the subjects, however, the improvement was temporary and decreased performance was seen again. Behavioral observations on each of these four subjects showed an increased tendency to cling to the lever and lean against the wall during the post-burn period.

Two subjects showed an apparent muscular weakness, losing their grip on the lever easily and having to support themselves by trying to hold the lever. None appeared to lose awareness of shock. In each case, the burn period produced an unusually high activity level with escape attempts. Lever pressing, however, continued and only began to subside after the oven was removed. This was the only sample which resulted in an animal's failure in his attempt to press the lever.

Sample Y-7212 was assigned to Group IV. There were no significant changes in avoidance rate for this group as a result of experimental treatment. Behavioral observations indicated little effect from pyrolysate exposure with the exception of one subject whose response was one of decreased activity and increased respiration rate. He appeared to be somewhat dazed. Data showed that his response rate did not improve during the last thirty minute interval as was typical of previous controls.

Sample Y-7191 was assigned to Group V. This group, unlike all others showed a significant difference between Day 1 and Day 3 in the direction of improvement, as well as a significant improvement on the third day from pre-burn and post-burn periods. Since the tendency was for all animals to improve both over first two days and over time on each of the days, the results for Group V are interpreted as indicating that Sample Y-7191 had the least effect on avoidance behavior of the 6 samples included in the statistical analyses. That is, it was the only sample that allowed the improvement by the end

of the three day testing period that would have been expected had not the experimental treatment been initiated. Behavioral observations indicated little response to the pyrolysis condition, with the exception of a startle reaction when the material flashed as burning commenced.

Sample Y-7213 was assigned to Group VI. For this sample, 37 gm. was used as the estimated LD_{50} , and one-half of this quantity (18.5 gm) was employed in the behavioral studies. No significant differences resulting from the treatment condition were found for this group. Only one subject showed a behavioral effect; however, this effect was profound. During the burn period his behavior became frantic and he would alternate escape behavior with lever pressing. By 5 minutes post-burn, he had completely stopped lever pressing and did not resume it. Although he often responds to the shock by squealing, he ignored it as often, and divided his time between grooming and attempting to climb out of the box.

Other Fractions of LD_{50} 's. An additional group (Group II) was created to test the effects of $1/4$ the LD_{50} of sample Y-7192, which had produced the most profound behavioral effects at $1/2$ the LD_{50} . This group was included in the statistical analysis with the five groups representing separate samples. Results showed that there was a significant difference in performance for this group between the first and third day during the post-burn period, and between the second and third day during that period, although no difference existed between the two (control) days. The profound difference resulting from sample pyrolysis seen in Group I were

not observed in the data for Group II. However all animals decreased in rate of response during the post-burn period. Behavioral observations showed a decrease in activity with apparent weakness, but no apparent insensitivity to shock or immobilization.

In addition to these groups, several other animals were tested on fractions of the LD₅₀ greater or less than one-half. All of these animals were trained and tested in the same manner as those comprising the groups subjected to statistical analysis.

Sample Y-7212 was tested at 1/4 LD₅₀ on two subjects. Although one animal showed a slight decrease in avoidance rate during the first 10 minute post-burn, both showed overall improvement during the post-burn period compared to the pre-burn period. No behavioral changes were noted.

Sample Y-7214 was tested on 3 subjects at 3/4 of the LD₅₀. Increasing the quantity of this sample produced an effect upon avoidance not seen at 1/2 LD₅₀, although the effect was variable. While one subject continued to respond at a rate comparable to that of the pre-burn period the second showed a slight but consistent decrease in responding. The third animal completely ceased avoidance behavior when the oven was removed and did not resume lever pressing. Behavioral observations indicate that although he generally ignored the shock he was not stuporous and divided his time between crouching, grooming and attempting to escape.

An additional subject was tested using the same procedure

as with the experimental animals, but with no pyrolysis sample introduced. In this case the oven was left in the chamber for 14 minutes and the chamber temperature was allowed to rise to 95°F. This subject showed improved performance from Days 1 and 2, and showed increased performance during the post-burn period. The average number of shocks received during the post-burn period was less than 0.8/min., one of the lowest shock rates found during any interval for any other subject. There was some increase in respiratory rate noted during the post-burn period but no other behavioral changes.

A ranking of these sample materials in order of their pyrolysis/combustion products' influence upon behavior (shock-avoidance) is presented in Table 20.

Concluding Comments

In the overall assessment of the relative safety of a candidate material for use in aircraft interiors, consideration should also be given to the thermodegradation (flaming or nonflaming degradation) characteristics of the material. Depending upon how and where the material is to be used the temperature required for initiation of degradation, the rate at which the material decomposes, and the maximum temperature required to complete its decomposition, may be significant factors. As an illustration, consider two foams included in this study: a polyimide (Y-7191) and a polyurethane (Y-7192).

The TGA data reveal that the polyimide (Y-7191) begins to degrade at a little over 500°C, with about 90% decomposed by 600°C in air, but only about 30% decomposition at 600°C when heated in nitrogen (suggesting oxidative thermodegradation is important in production of volatile products), with degradation being essentially complete, in air, by about 700°C. By comparison, the polyurethane foam (Y-7192) shows initial degradation at about 150°C and is essentially complete (about 97-98%) by 600°C, with little or no difference between an atmosphere of air or nitrogen. Examination of the toxicity data also reveal some interesting comparisons. Based upon initial weight of sample pyrolyzed, the polyimide foam is significantly more toxic, as suggested by lethality of rats exposed to the pyrolysis/combustion products. On the other hand the behavioral tests indicate that rats' response patterns are disrupted more by exposure to pyrolysis/combustion product generated by one-half of the LD₅₀ of the polyurethane foam sample than the polyimide foam sample. From the applications aspect another factor to consider is the density (weight/unit volume) of the two foams. Consequently, the weight of a functional unit (such as a seat cushion) may be markedly different for the two foams which would be important in regard to potential toxic hazard from an aircraft fire.

Thus in an assessment to select the "best" material, the data suggest that (1) the polyurethane foam (Y-7192) would be less likely to produce death from its pyrolysis/combustion than would an equal weight of the polyimide foam (Y-7191);

(2) on the other hand, the polyimide foam (Y-7191) requires a higher temperature for thermodegradation, has a lower density, and exposure to sublethal levels of pyrolysates exhibited less tendency to disrupt the behavior of trained rats.

Table 1
IDENTIFICATION OF SAMPLES
(Aircraft Materials)

Y-7191	Polyimide Foam
Y-7192	Polyurethane Foam
Y-7212	Orcon Kapton with Kel-F 800 with AB 312 Ceramic Filer and Nylon Fibers (subsequent sample identified as: "Orcon KN-80 C-22-1254E, lot no. 628")
Y-7213	3M Silicone on Fiberglass
Y-7214	Polyimide Foam, #720-1

Table 2
ANALYSIS OF TGA DATA

Y-7191 Polyimide Foam					
Identification	345	380	342	341	
TGA Run No.					
Atmosphere	Air	Air	Nitrogen	Nitrogen	
Flow Rate	200 ml/min	20 ml/min	200 ml/min	20 ml/min	
Heating Rate	10°C/min	20°C/min	10°C/min	20°C/min	
Sample Weight	1.7 mg	2.72 mg	2.3 mg	3.0 mg	
Approximate Initiation of Decomposition, °C*	535	385	475	480	
Approximate Completion of Decomposition, °C*	615	635	----	----	
Maximum TGA Temp.	845°C	924°C	987°C	1068°C	
Final Residue Weight	0 mg	0 mg	1.0 mg	1.3 mg	
Percent Final Residue	0%	0%	44%	46%	
Approximate Temperature for 50% Degradation, °C*	560	555	750	1010	
Percent Residue at 600°C	6.0%	14%	62%	75%	

380 is a repeat
TGA run of #337.

* Value to nearest 5°C.

Table 3
ANALYSIS OF TGA DATA

Identification	Y-7192 Greenish polyurethane foam				
TGA Run No.	346 & 344	338	343	339	
Atmosphere	Air	Air	Nitrogen	Nitrogen	
Flow Rate	200 ml/min	20 ml/min	200 ml/min	20 ml/min	
Heating Rate	10°C/min	20°C/min	10°C/min	20°C/min	
Sample Weight	5.1 g & 5.4 mg	4.3 mg	3.85 mg	5.1 mg	
Approximate Initiation of Decomposition, °C*	184 & 140	115	120	170	
Approximate Completion of Decomposition, °C*	720 & 540	675	380	775	
Maximum TGA Temp.	807 & 968°C	999°C	957°C	975°C	
Final Residue Weight	0 & 0 mg	0 mg	0 mg	0 mg	
Percent Final Residue	0 & 0%	0%	0%	0%	
Approximate Temperature for 50% Degradation, °C*	260 & 280	245	345	325	
Percent Residue at 600°C	1.8 & 2.8%	1.0%	6.0%	4.0%	

* Value to nearest 5°C.

Table 4
ANALYSIS OF TGA DATA

Identification	Y-7212 composite material	(Orcon Kapton with Kel-F with AB 312 Ceramic Filer & Nylon Fibers)	
TGA Run No.	367	373	363
Atmosphere	Air	Air	Nitrogen
Flow Rate	200 ml/min	20 ml/min	20 ml/min
Heating Rate	10°C/min	20°C/min	20°C/min
Sample Weight	7.15 mg	7.45 mg	5.60 mg
Approximate Initiation of Decomposition, °C*	300	300	340
Approximate Completion of Decomposition, °C*	605	850	810
Maximum TGA Temp.	731°C	971°C	1019°C
Final Residue Weight	0 mg	1.45 mg	2.26 mg
Percent Final Residue	0%	20%	40%
Approximate Temperature for 50% Degradation, °C*	545	585	700
Percent Residue at 600°C	0.4%	46%	58%

* Value to nearest 5°C.

Table 5
ANALYSIS OF TGA DATA

Identification	Y-7213 3M Silicone on Fiberglass			
TGA Run No.	368 & 349	350 & 348	362	
Atmosphere	Air	Air	Nitrogen	
Flow Rate	200 ml/min	20 ml/min	20 ml/min	
Heating Rate	10°C/min	20°C/min	20°C/min	
Sample Weight	5.76 & 10.0 mg	9.66 & 16.38 mg	8.51 mg	
Approximate Initiation of Decomposition, °C*	370 & 340	340 & 335	350	
Approximate Completion of Decomposition, °C*	790 & ---	750 & ---	765	
Maximum TGA Temp.	1200 & 882 °C	881 & 946°C	993°C	
Final Residue Weight	3.96 & 7.7 mg	6.96 & 12.6 mg	6.21 mg	
Percent Final Residue	69.0 & 77.0%	72.0 & 77%	73%	
Approximate Temperature for 50% Degradation, °C*	---- & ----	---- & ----	----	
Percent Residue at 600°C	80.0 & 79.5%	74.4 & 78.4%	74.9%	

* Value to nearest 5°C.

Table 6
ANALYSIS OF TGA DATA

Y-7214 Polyimide Foam #720-1				
Identification	376	352 & 347	361	
TGA Run No.				
Atmosphere	Air	Air	Nitrogen	
Flow Rate	200 ml/min	20 ml/min	20 ml/min	
Heating Rate	10°C/min	20°C/min	20°C/min	
Sample Weight	2.78 mg	1.45 & 2.45 mg	2.38 mg	
Approximate Initiation of Decomposition, °C*	350	325 & 370	405	
Approximate Completion of Decomposition, °C*	565	625 & 720	785	
Maximum TGA Temp.	691°C	722 & 941	1013	
Final Residue Weight	0 mg	0 & 0 mg	0.58 mg	
Percent Final Residue	0%	0 & 0%	24%	
Approximate Temperature for 50% Degradation, °C*	515	530 & 575	670	
Percent Residue at 600°C	0%	1.4 & 8.6%	58%	

* Value to nearest 5°C.

Table 7
LD₅₀ AND (95% CONFIDENCE INTERVAL) BASED UPON INITIAL
WEIGHT OF SAMPLE PYROLYZED

Sample	Chamber	48 Hours	14-Days
Y-7191 Polyimide Foam	2.63 gm (2.49-2.78)	2.56 gm (2.40-2.73)	2.56 (2.40-2.73)
Y-7192 Polyurethane Foam	6.77 gm (6.55-6.99)	6.77 gm (6.55-6.99)	6.23 gm (-----)
Y-7212 Orcon Kapton with Kel-F 800 with AB 312 Ceramic Filer and nylon fibers	4.11 gm (3.78-4.48)	4.11 gm (3.78-4.48)	4.00 gm (3.75-4.27)
Y-7213 3M Silicone on Fiberglass	Insufficient quantity of sample to complete LD ₅₀ determination; no deaths were produced by 25 gm and less, while 45 gm killed all of the rats during chamber exposure.		
Y-7214 Polyimide Foam, #720-1	4.86 gm (4.47-5.29)	4.86 gm (4.47-5.29)	4.86 gm (4.47-5.29)

Table 8

LETHALITY TOXICITY DATA AND PERCENT THERMODEGRADATION OF SAMPLES

Sample	14-Day LD ₅₀ *	Theoretical % Decomposition (from TGA)**		Theoretical Sample Weight Decomposed at LD ₅₀	
		est. @ 700°C	Final (900-1000°C)	@ 700°C	Final
Y-7191	2.56 gm	~94%	100%	~2.4 gm	2.56 gm
Y-7192	6.23 gm	~99%	100%	~6.2 gm	6.23 gm
Y-7212	4.00 gm	~67%	80%	~2.7 gm	3.20 gm
Y-7213	# >25 gm; <45 gm	~28%	30%***	>7.0 <12.6 gm#	>7.5 <13.5 gm#
Y-7214	4.86 gm	~100%	100%	~4.86 gm	4.86 gm

* Chamber volume is approximately 200 liters.

** TGA determined in air, with an air flow rate of 20 ml/min, and heating rate of 20°C/min.

*** The actual percent of sample decomposed in the pyrolysis-inhalation experiments was determined to be 25-28% of initial sample weight.

The LD₅₀ value was not determined because of the limited supply of sample; available quantity of sample was used for behavioral tests. Initial sample weights of 25 gm or less did not kill any of the exposed rats, while a 45 gm sample killed all of the exposed rats.

Table 9

RANKING OF SAMPLES BY LETHALITY* FROM EXPOSURE
TO PYROLYSIS/COMBUSTION PRODUCTS

I. Based Upon Initial Weight of Sample

Least Toxic	Y-7213 3M Silicone on Fiberglas**
	Y-7192 Polyurethane Foam
	Y-7214 Polyimide Foam, #720-1
	Y-7212 Orcon Kapton with Kel-F 800 with AB 312 Ceramic Filer and Nylon Fibers
Most Toxic	Y-7191 Polyimide Foam

II. Based Upon Quantity of Sample Pyrolyzed/Combusted

Least Toxic	Y-7213 3M Silicone on Fiberglas***
	Y-7192 Polyurethane Foam
	Y-7214 Polyimide Foam, #720-1
	Y-7212 Orcon Kapton with Kel-F 800 with AB 312 Ceramic Filer and Nylon Fibers
Most Toxic	Y-7191 Polyimide Foam

* Although some rats died after removal from the exposure chamber, the changes were sufficient to alter the ranking of samples between chamber deaths and 14-day post-exposure periods.

** The limited quantity of this sample precluded completion of LD₅₀ determination, however data indicated it to be greater than 25 gm and less than 45 gm.

*** This, of necessity, is an estimate, but based upon 25% of the sample being pyrolyzed during heating and the LD₅₀ being somewhat greater than 25 gm, this seems to be a reasonable assumption.

Table 10

ACUTE TOXICITY OF PYROLYSIS/COMBUSTION PRODUCTS

Sample Y-7191 Polyimide Foam

Initial Weight	Percent Pyrolyzed	Mortality		--- Chamber Atmosphere Analysis*---				
		Chamber	Delayed (mean)	COHb	O ₂	CO ₂	CO	H ₂ O (mg/L)
2.05 gm	0%	0%	0%	N/A	(19%)	(1.51%) 1%	(0.11%) 0.1%	
2.29 gm	0%	0%	0%	N/A	(19%)	(2.71%) 1%	(0.16%) 0.1%	18
2.56 gm	25%	25%	25%	63%	(18%)	(1.37%) 0.9%	(0.21%) 0.2%	30
2.86 gm	100%	100%	0%	65%	(17%)	(2.32%) -	(0.31%) 0.4%	>18
3.20 gm	100%	100%	0%	70%	(18%)	(2.69%) 1.25%	(0.21%) 0.25%	

* Gas chromatographic analyses were conducted immediately after pyrolysis of sample (0 time), and after 15 and 30 minutes. These are reported as the mean of the three determinations enclosed in parentheses. Other values were obtained from detector tubes.

Table 11

ACUTE TOXICITY OF PYROLYSIS/COMBUSTION PRODUCTS

Sample: Y-7192 Polyurethane Foam

Initial Weight	Percent Pyrolyzed	Mortality		COHb (mean)	---- Chamber Atmosphere Analysis* ----				
		Chamber	Delayed		O ₂	CO ₂	CO	HCN (ppm)	H ₂ O (mg/L)
5.72 gm		0%	0%	N/A	(18%)	(1.29%) 0.25%	(0.10%) 0.1%	10	
6.05 gm		0%	0%	N/A	(18%)	(0.97%) 0.9%	(0.15%) 0.1%	10	2
6.40 gm		0%	100%	N/A	(18%)	(1.28%) 1.0%	(0.19%) 0.15%	40	3
6.77 gm		100%	0%	65%	(17%)	(2.22%) 1.25%	(0.27%) 0.2%	30	3
7.15 gm		50%	50%	63%	(18%)	(0.95%) 1.5%	(0.22%) 0.35%	>60	
7.57 gm		100%	0%	79%	(19%)	(0.88%) 0.9%	(0.31%) 0.22%	60	3

* Gas chromatographic analyses were conducted immediately after pyrolysis of sample (0 time), and after 15 and 30 minutes. These are reported as the mean of the three determinations enclosed in parentheses. Other values were obtained from detector tubes.

Table 12

ACUTE TOXICITY OF PYROLYSIS/COMBUSTION PRODUCTS

Sample Y-7212 Orcon Kapton with Kel-F 800 with AB 312 Ceramic Filer and Nylon Fibers

---Chamber Atmosphere Analysis*---									
Initial Weight	Percent Pyrolyzed	Mortality		CO ₂ b	O ₂	CO ₂	CO	HCN (ppm)	H ₂ O (mg/L)
		Chamber	Delayed						
3.20 gm	64%	0%	0%	n/a	(20%)	(1.3%) 1.3%	(0.13%) 0.2%	>60	14
3.58 gm	62%	0%	0%	n/a	(20%)	(1.1%) 1.2%	(0.1%) 0.18%	>60	12
4.00 gm	74%	50%	0%	59%	(20%)	(2.0%) 1.7%	(0.14%) 0.2%	>60	16
4.47 gm	70%	75%	25%	67%	(19.5%)	(1.9%) 1.8%	(0.13%) 0.2%	>60	18
5.00 gm	71%	100%	0%	57%	(19%)	(1.3%) 1.8%	(0.18%) 0.25%	>60	>18

* Gas chromatographic analyses were conducted immediately after pyrolysis of sample (0 time), and after 15 and 30 minutes. These are reported as the mean of the three determinations enclosed in parentheses. Other values were obtained from detector tubes.

Table 13

ACUTE TOXICITY OF PYROLYSIS/COMBUSTION PRODUCTS

Sample Y-7213 3M Silicone on Fiberglass

---Chamber Atmosphere Analysis*---									
Initial Weight	Percent Pyrolyzed	Mortality		COHb (mean)	O ₂	CO ₂	CO	HCN (ppm)	H ₂ O (mg/L)
		Chamber	Delayed						
5.00 gm	25%	0%	0%	n/a	(19%)	(0.31%) 0.6%	(0.03%) 0.03%	2	---
25.00 gm	27%	0%	0%	n/a	(19%)	(1.92%) 1.9%	(0.10%) 0.13%	15	---
45.00 gm	28%	100%	0%	56%	(17%)	(4.10%) 4.0%	(0.11%) 0.10%	1	---

* Gas chromatographic analyses were conducted immediately after pyrolysis of sample (0 time), and after 15 and 30 minutes. These are reported as the mean of the three determinations enclosed in parentheses. Other values were obtained from detector tubes.

Table 14

ACUTE TOXICITY OF PYROLYSIS/COMBUSTION PRODUCTS

Sample Y-7214 Polyimide Foam, #720-1

--- Chamber Atmosphere Analysis* ---									
Initial Weight	Percent Pyrolyzed	Mortality		COHb (mean)	O ₂	CO ₂	CO	HCN (ppm)	H ₂ O (mg/L)
		Chamber	Delayed						
4.00 gm	87%	0%	0%	N/A	(20%)	(1.30%) 2.3%	(0.14%) 0.21%	>60	16
4.47 gm	84%	25%	0%	72%	(19%)	(2.12%) 2.7%	(0.17%) 0.28%	>60	17
5.00 gm	79%	50%	0%	65%	(19%)	(2.30%) 2.8%	(0.17%) 0.3%	>60	>18
5.59 gm	82%	100%	0%	80%	(19%)	(2.35%) 3.0%	(0.22%) 0.3%	>60	>18

* Gas chromatographic analyses were conducted immediately after pyrolysis of sample (0 time), and after 15 and 30 minutes. These are reported as the mean of the three determinations enclosed in parentheses. Other values were obtained from detector tubes.

Table 15

SUMMARY OF HISTOPATHOLOGIC EVALUATION

Sample: Y- 7191 Polyimide Foam

Histopathologic features observed in organs/tissue of rats exposed to pyrolysis/combustion products of this material suggest inhalation of these products produced the following:

5/10 animals showed pulmonary congestion acute, focal & diffuse, moderate to severe. The other 5/10 animals showed chronic pulmonary disease such as bronchopneumonia focal, chronic moderate to severe & pneumonia chronic focal mild & 1 of which were not related to inhalation exposure of the rats to the pyrolysates.

Histopathologic features observed in organs/tissues from rats exposed to pyrolysis/combustion products of this material suggest the following are delayed reactions (pathologies) which resulted from such exposure:

5/6 animals showed vasculitis, chronic focal or diffuse, mild to moderate. 1 of the 5 (1/5) also showed acute massive hemorrhage, 1/5 showed pneumonia chronic, focal mild, 1/5 showed pneumonia chronic focal mild and bronchopneumonia focal chronic moderate. The remaining 1/6 animal showed pneumonia, chronic, focal, mild. Much granular debris was noted in alveolar spaces.

(Note! This summary should exclude any abnormality which does not appear to be related to inhalation exposure of the rats to the pyrolysates.)

Table 16

SUMMARY OF HISTOPATHOLOGIC EVALUATION

Sample: Y-7192 Polyurethane Foam

Histopathologic features observed in organs/tissue of rats exposed to pyrolysis/combustion products of this material suggest inhalation of these products produced the following:

4/8 animals showed evidence of chronic pulmonary disease
the other 4/8 animals showed pulmonary congestion and edema
acute diffuse, mild to severe

& NOT RELATED TO INHALATION EXPOSURE TO PYROLYSATES

Histopathologic features observed in organs/tissues from rats exposed to pyrolysis/combustion products of this material suggest the following are delayed reactions (pathologies) which resulted from such exposure:

3/5 animals showed Bronchopneumonia, CHRONIC
FOCAL TO DIFFUSE MODERATE TO MASSIVE

2/5 animals showed ^{PNEUMONITIS} CHRONIC FOCAL MILD TO MODERATE

ORIGINAL PAGE IS
OF POOR QUALITY

(Note! This summary should exclude any abnormality which does not appear to be related to inhalation exposure of the rats to the pyrolysates.)

Table 17

SUMMARY OF HISTOPATHOLOGIC EVALUATION

Sample: Y- 7212 Orion Kapton with Kel-F 800 with AB 312
Ceramic Filer and Nylon Fibers

Histopathologic features observed in organs/tissue of rats exposed to pyrolysis/combustion products of this material suggest inhalation of these products produced the following:

2/4 animals showed congestion and edema
acute, diffuse, mild and 1 of these 2 (1/2)
showed hemorrhage acute, focal, moderate
the other 2/4 showed evidence of
chronic pulmonary disease which was not
related to inhalation exposure of the rats but
did contribute to the animals death.

Histopathologic features observed in organs/tissues from rats exposed to pyrolysis/combustion products of this material suggest the following are delayed reactions (pathologies) which resulted from such exposure:

8/9 animals showed pneumonia, chronic, focal
mild to moderate. 3 of these 8 (3/8) also showed
vasculitis, focal, chronic, mild to moderate.
1/9 showed only congestion and edema, acute
diffuse mild

(Note! This summary should exclude any abnormality which does not appear to be related to inhalation exposure of the rats to the pyrolysates.)

Table 18

SUMMARY OF HISTOPATHOLOGIC EVALUATION

Sample: Y- 7213 3M Silicone on Fiberglass

Histopathologic features observed in organs/tissue of rats exposed to pyrolysis/combustion products of this material suggest inhalation of these products produced the following:

*2/2 ANIMALS SHOWED EVIDENCE OF MASSIVE
INSPIRATION OF PYROLYSIS DEBRIS*

Histopathologic features observed in organs/tissues from rats exposed to pyrolysis/combustion products of this material suggest the following are delayed reactions (pathologies) which resulted from such exposure:

*3/3 ANIMALS SHOWED PNEUMONITIS, CHRONIC
FOCAL, MILD TO MODERATE AND/OR VASCULITIS CHRONIC
FOCAL MILD.*

(Note! This summary should exclude any abnormality which does not appear to be related to inhalation exposure of the rats to the pyrolysates.)

(PT 0.1737)

-47-

Table 19

SUMMARY OF HISTOPATHOLOGIC EVALUATION

Sample: Y-7214 Polyimide Foam #726-1

Histopathologic features observed in organs/tissue of rats exposed to pyrolysis/combustion products of this material suggest inhalation of these products produced the following:

NO FEATURES DUE TO PYROLYSIS/COMBUSTION PRODUCTS ONLY SINCE 4/4 ANIMALS SHOWED EVIDENCE OF CHRONIC PULMONARY DISEASE (NOT DUE TO PYROLYSIS) SUCH AS VASCULITIS, CHRONIC, FOCAL MODERATE; BRONCHOPNEUMONIA CHRONIC, FOCAL, MILD. PNEUMONITIS, CHRONIC, FOCAL M.C.D.

Histopathologic features observed in organs/tissues from rats exposed to pyrolysis/combustion products of this material suggest the following are delayed reactions (pathologies) which resulted from such exposure:

4/7 ANIMALS SHOWED BRONCHOPNEUMONIA CHRONIC, FOCAL, MILD TO SEVERE. 2 OF THESE 4 (2/4)^{also} SHOWED BRONCHITIS ACUTE AND CHRONIC SEVERE. 1/4 SHOWED PERIBRONCHITIS, CHRONIC MASSIVE. 1/4 SHOWED CHRONIC TRACHEITIS SEVERE.

OF THE REMAINING ANIMALS 1/3 SHOWED

BRONCHITIS, ACUTE AND CHRONIC, SEVERE, 1/3 SHOWED

FOCAL TRACHEAL EROSION AND 1/3 SHOWED PNEUMONITIS

FOCAL, CHRONIC, MODERATE TO SEVERE AND CONGESTION AND EDEMA ACUTE DIFFUSE, MILD TO SEVERE

(Note! This summary should exclude any abnormality which does not appear to be related to inhalation exposure of the rats to the pyrolysates.)

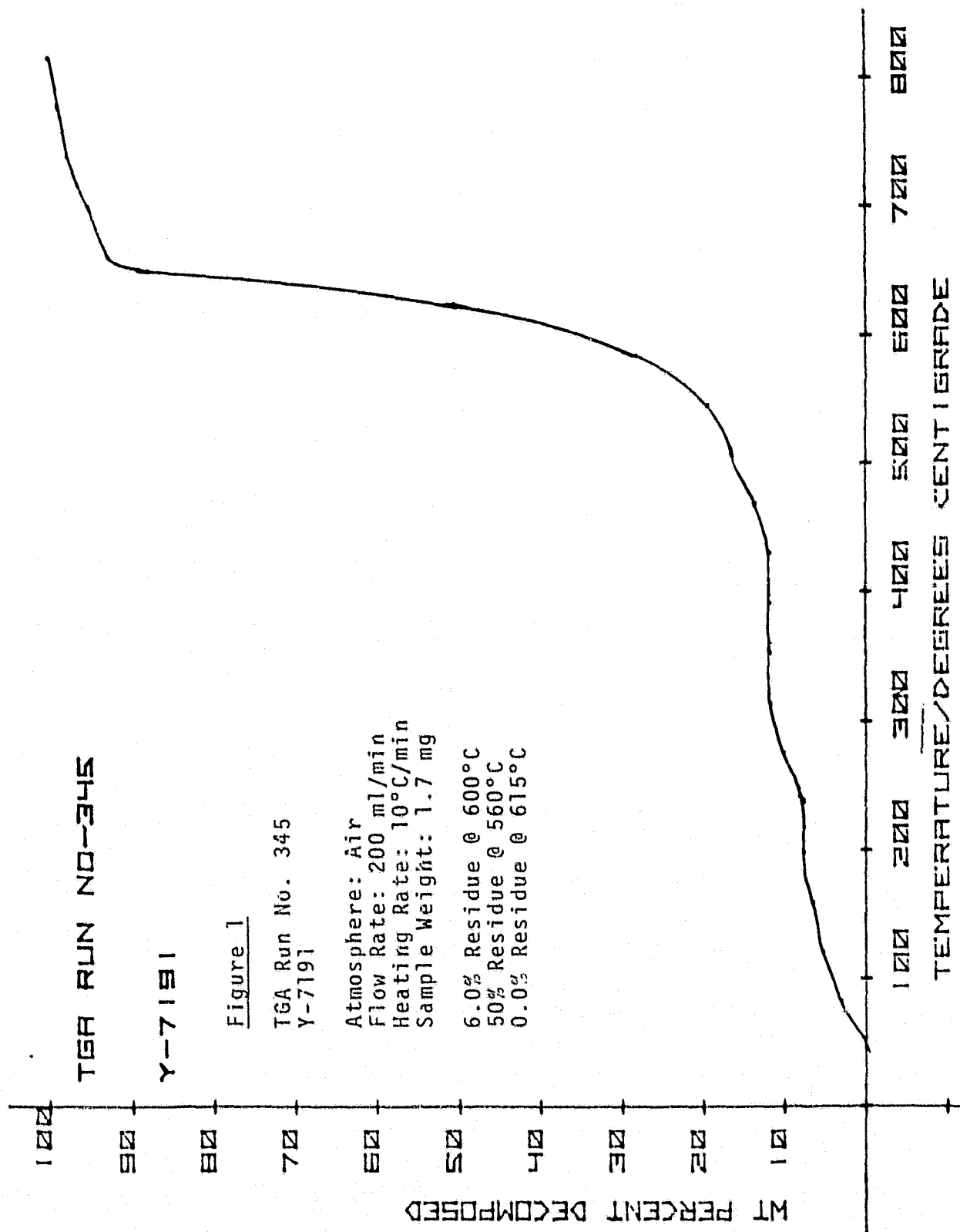
Table 20

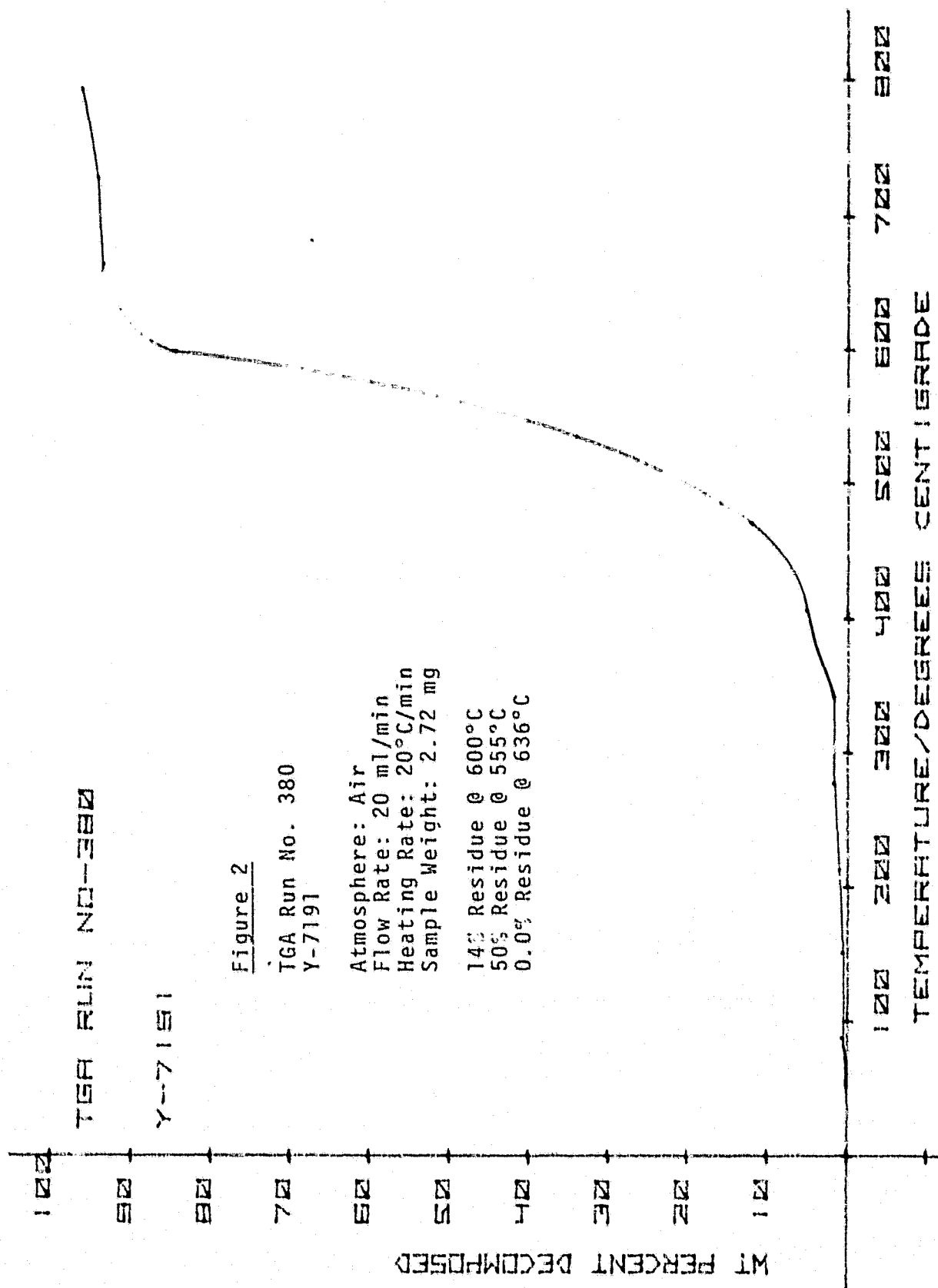
RANKING OF SAMPLES BASED UPON TENDENCY FOR
PYROLYSIS/COMBUSTION PRODUCTS FROM ONE-HALF
OF LD₅₀ OF SAMPLE TO INTERFERE WITH BEHAVIORAL
(SHOCK-AVOIDANCE) PERFORMANCE OF RATS*

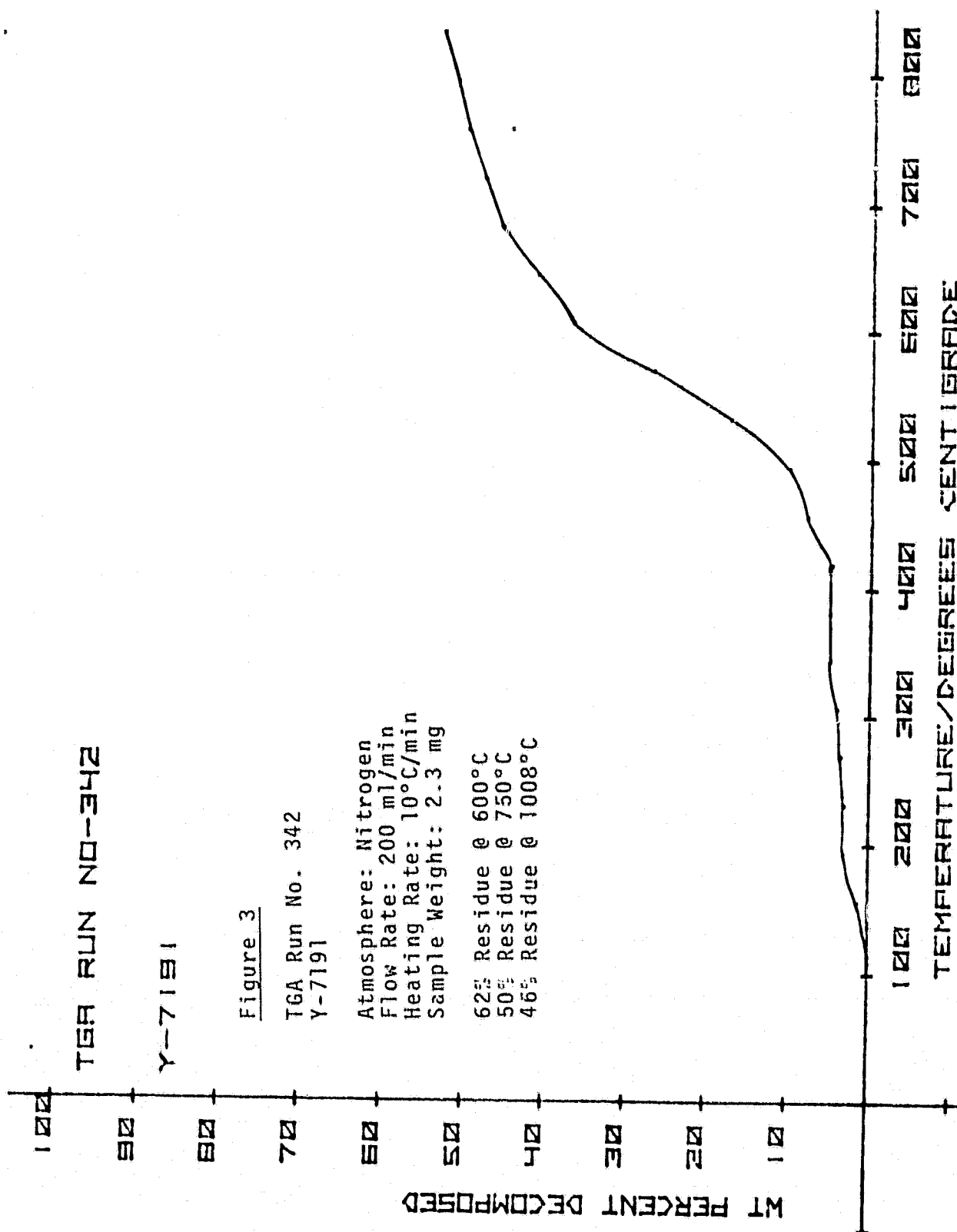
Least Effect	Y-7191 Polyimide Foam
	Y-7212 Orcon Kaptom with Kel-F
	** Y-7213 3M Silicone on Fiberglass
	Y-7214 Polyimide Foam, #720-1
Greatest Effect	Y-7192 Polyurethane Foam

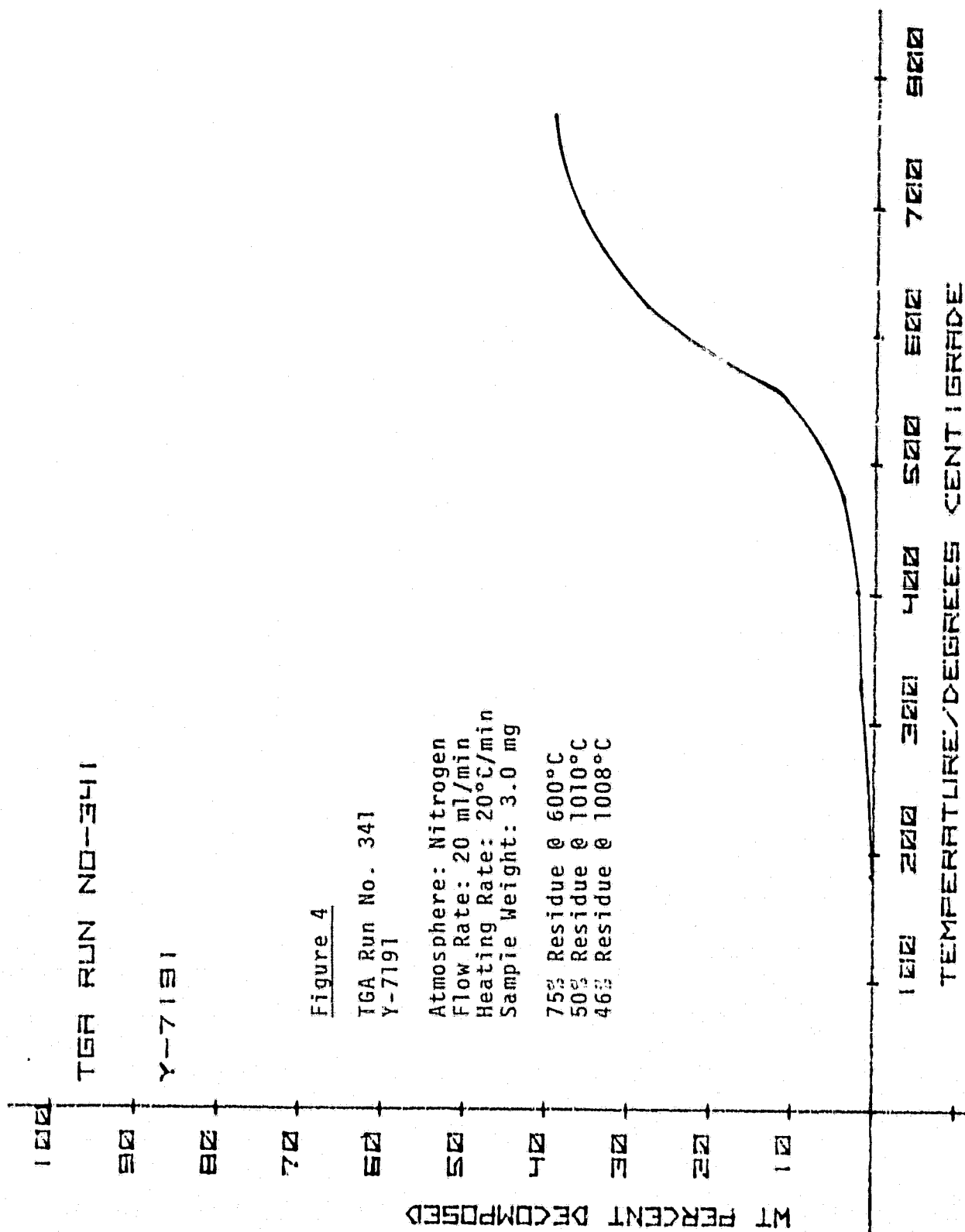
* At 1/2 of the LD₅₀ sample size, only Y-7192 showed a statistically significant behavioral difference in the direction of a decreased performance. Y-7214 did show a statistically significant decrease in performance in the first time interval following sample pyrolysis. Ranking of the other samples was based upon general trends of shock-avoidance activity and upon general observations of the exposed rats.

** For this study a LD₅₀ of 37 gm was arbitrarily assigned to Y-7213. As mentioned in other portions of this report there was not enough of this sample to complete the LD₅₀ study and still do the behavioral study. Initial data, however, indicated the LD₅₀ to be between 25 gm and 45 gm, since pyrolysis of 25 gm did not kill any of the exposed rats, while 45 gm killed all of them.

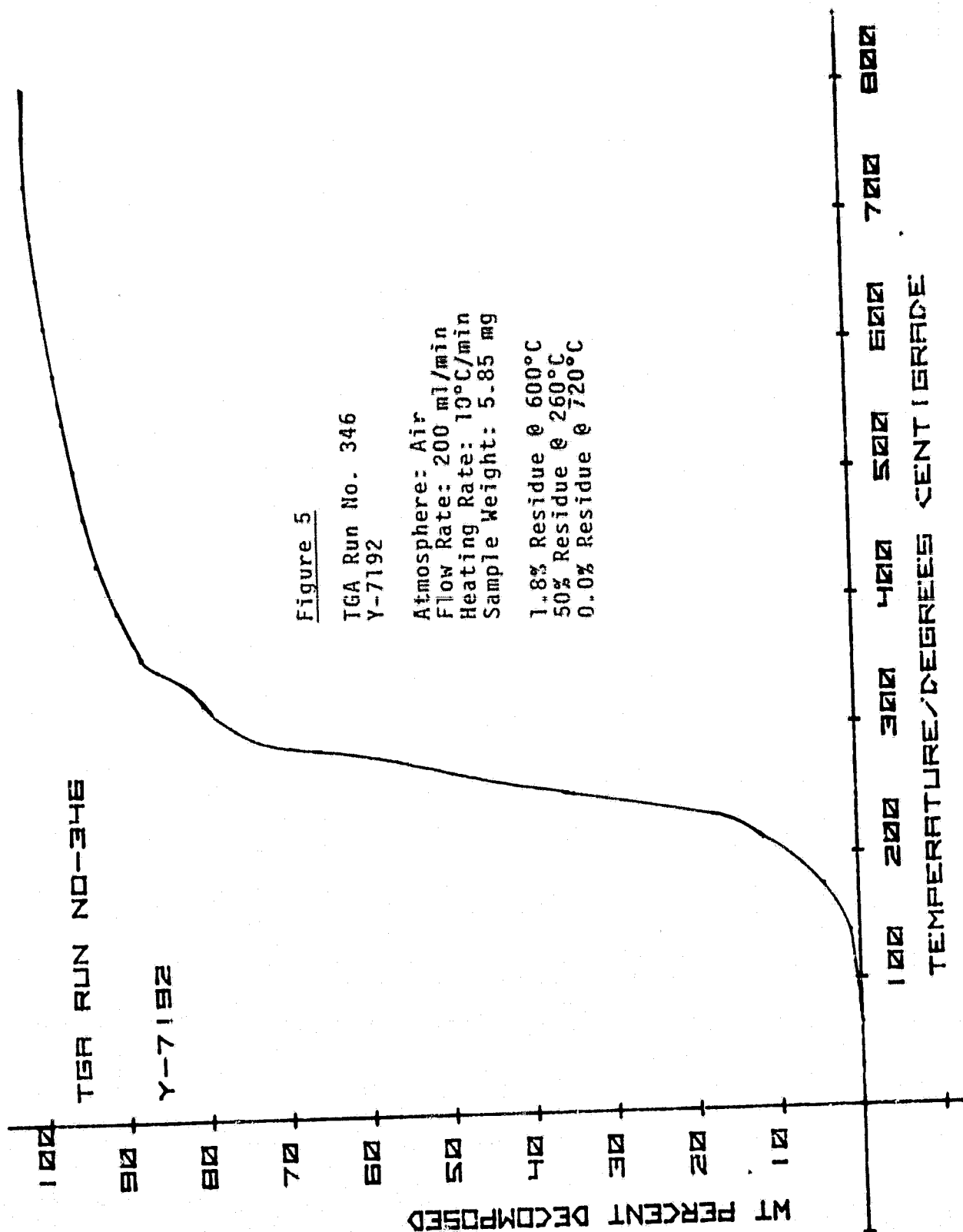


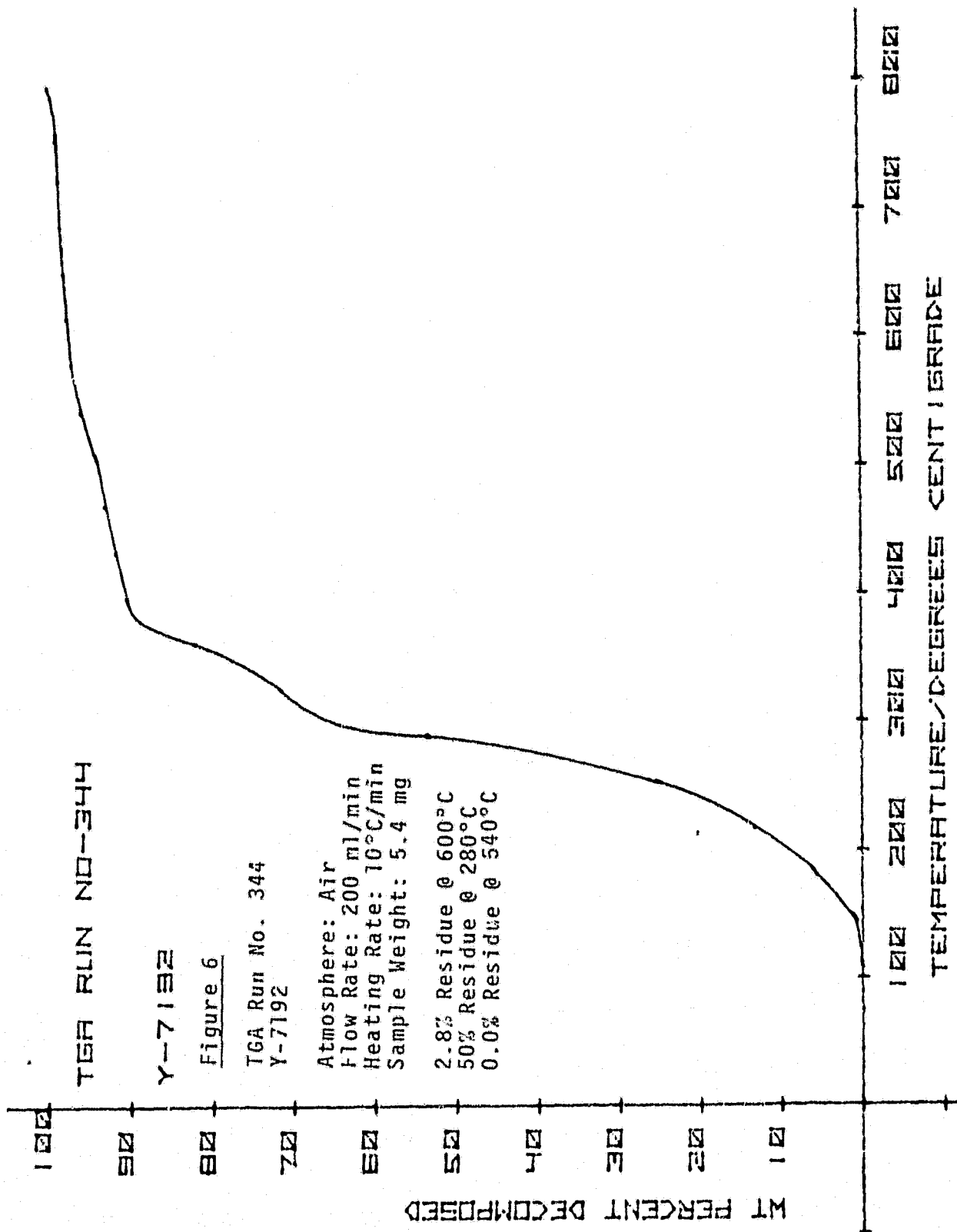


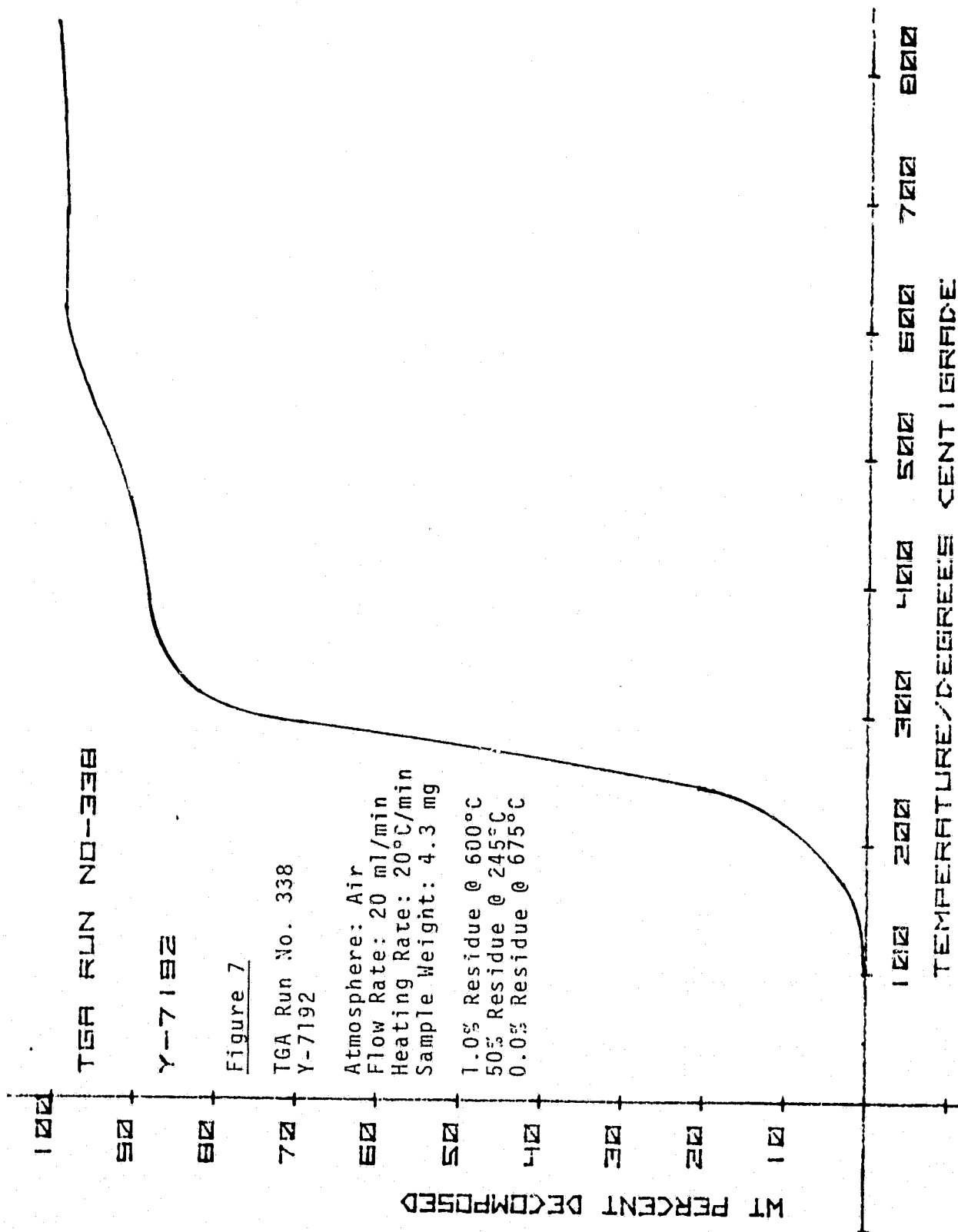


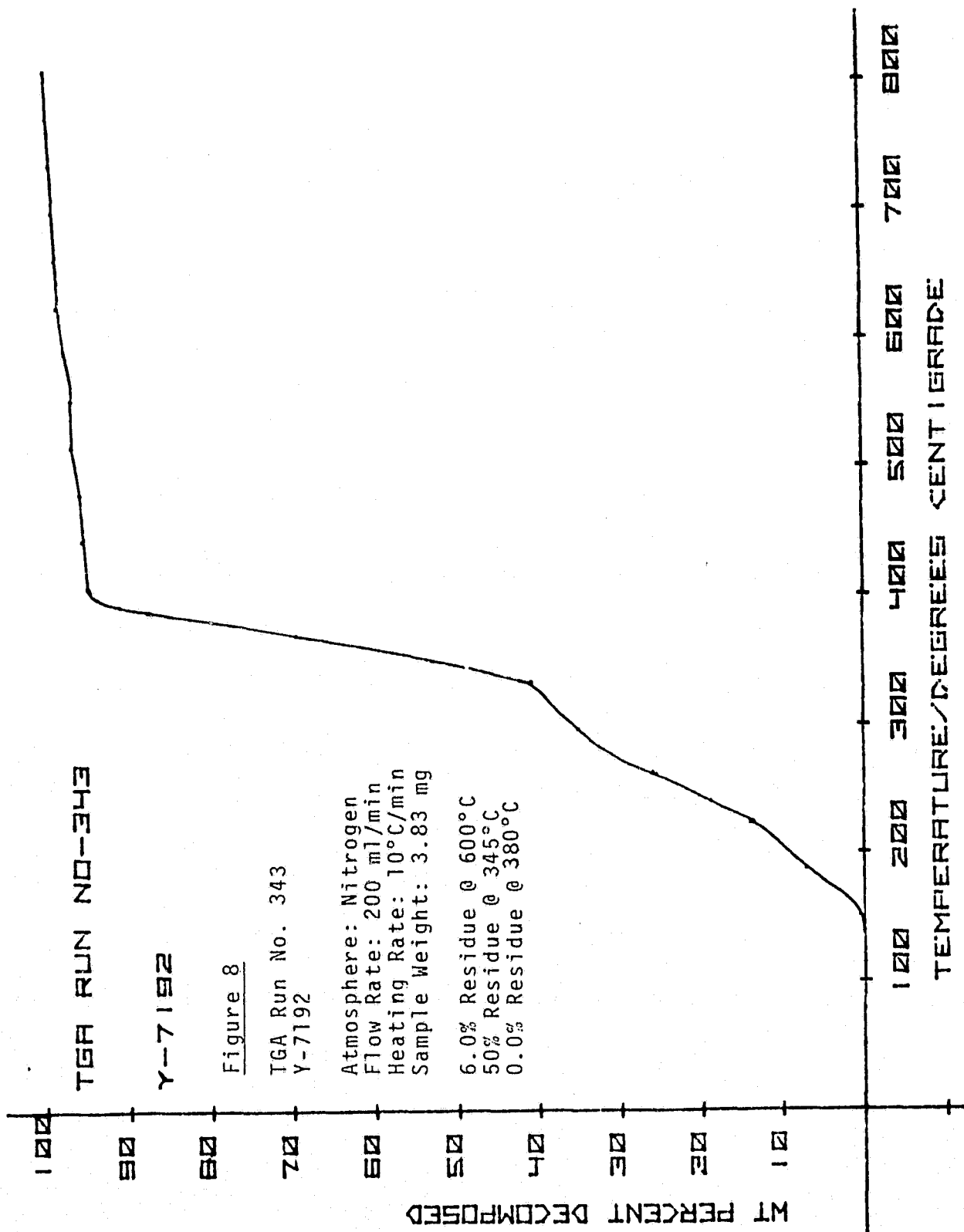


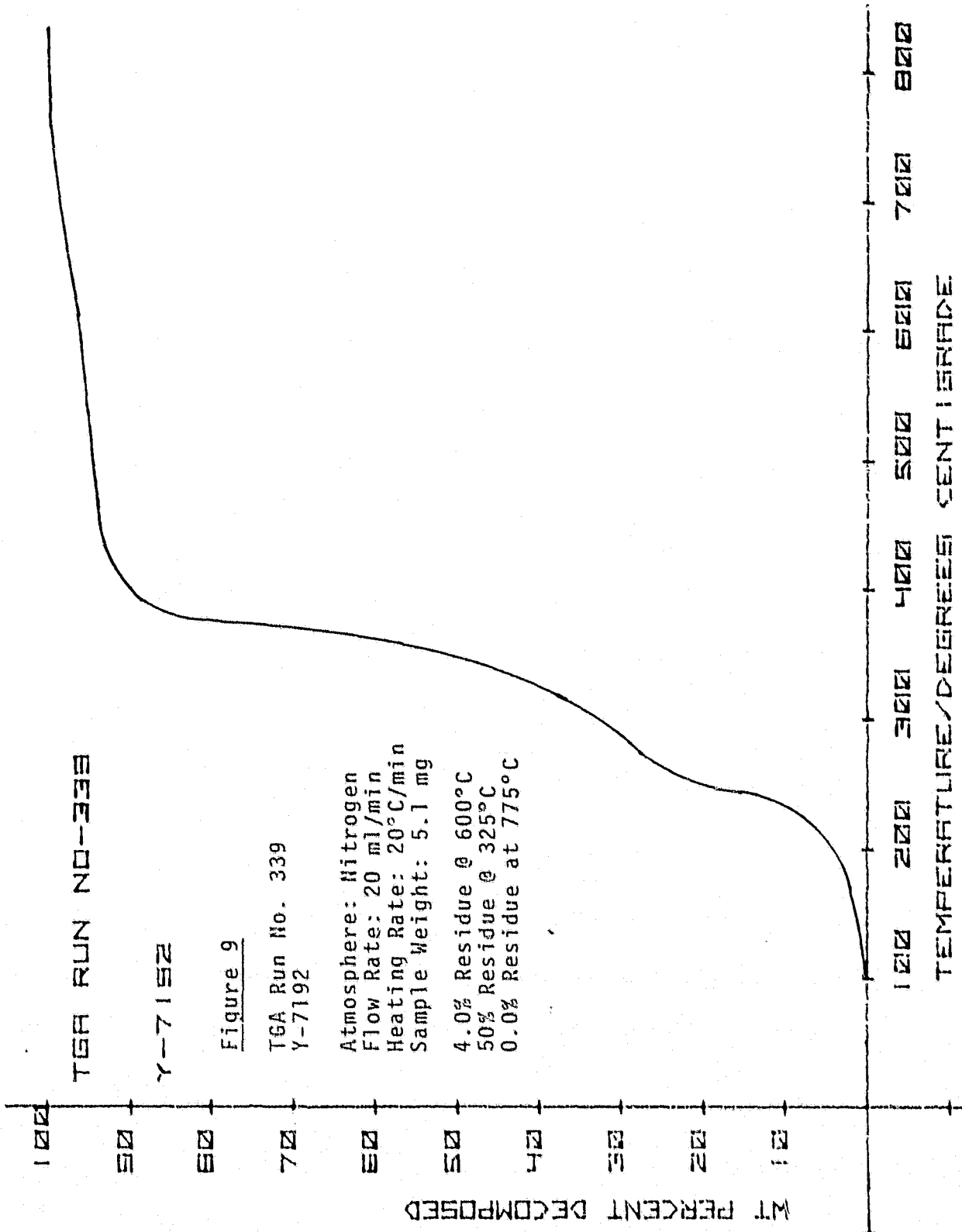
ORIGINAL PAGE IS
OF POOR QUALITY

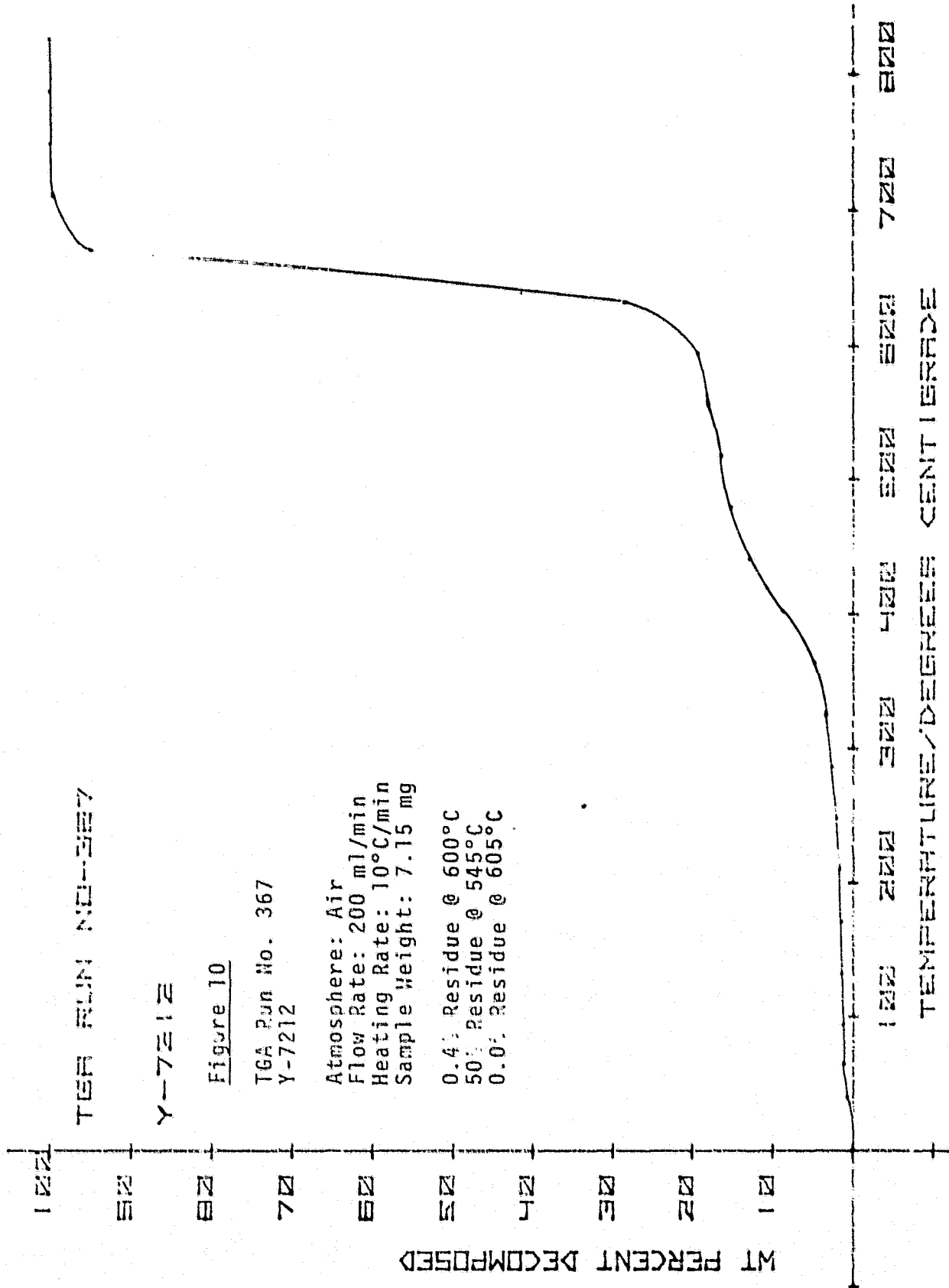












TER RUN NO-373

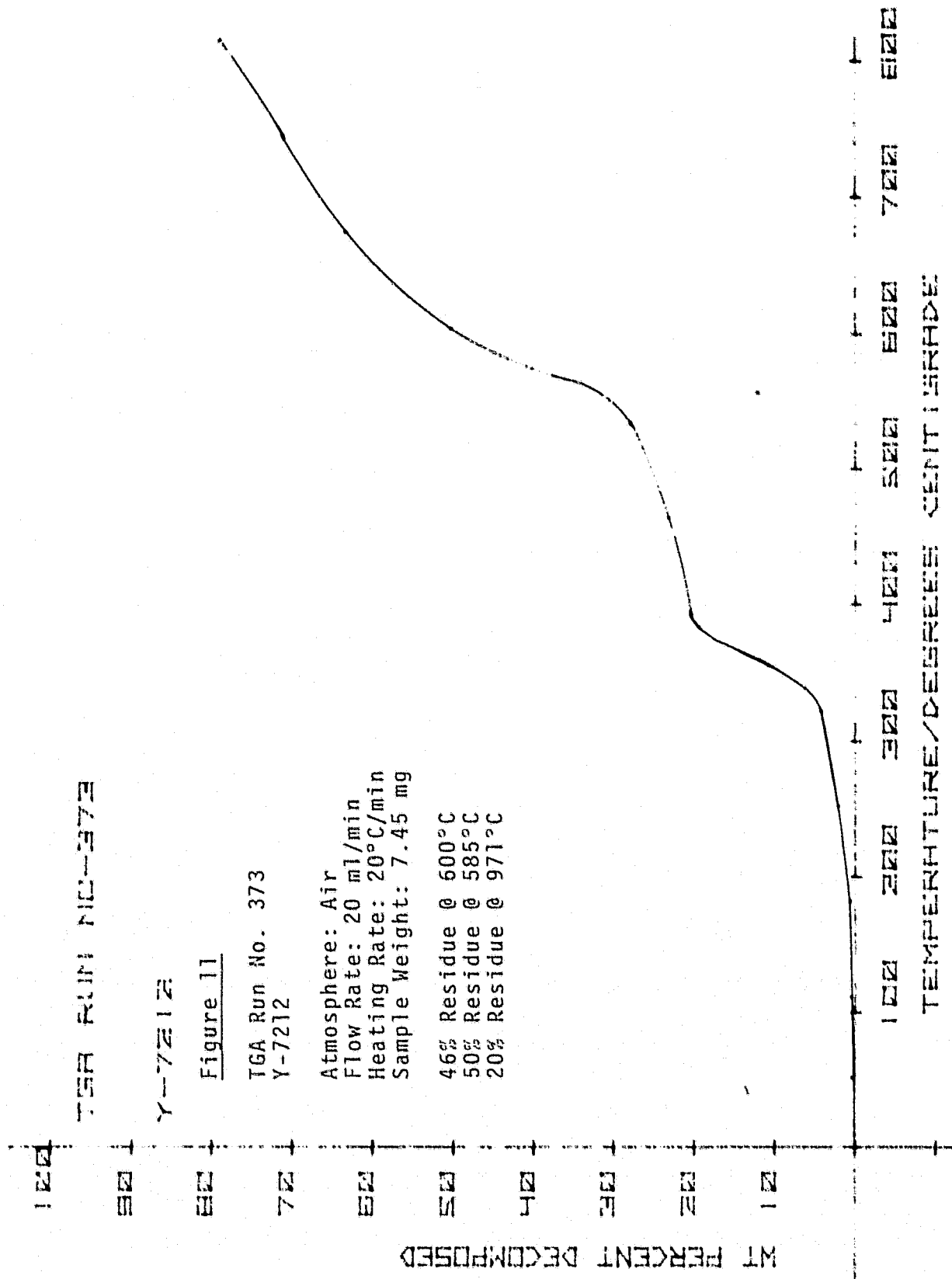
Y-7212

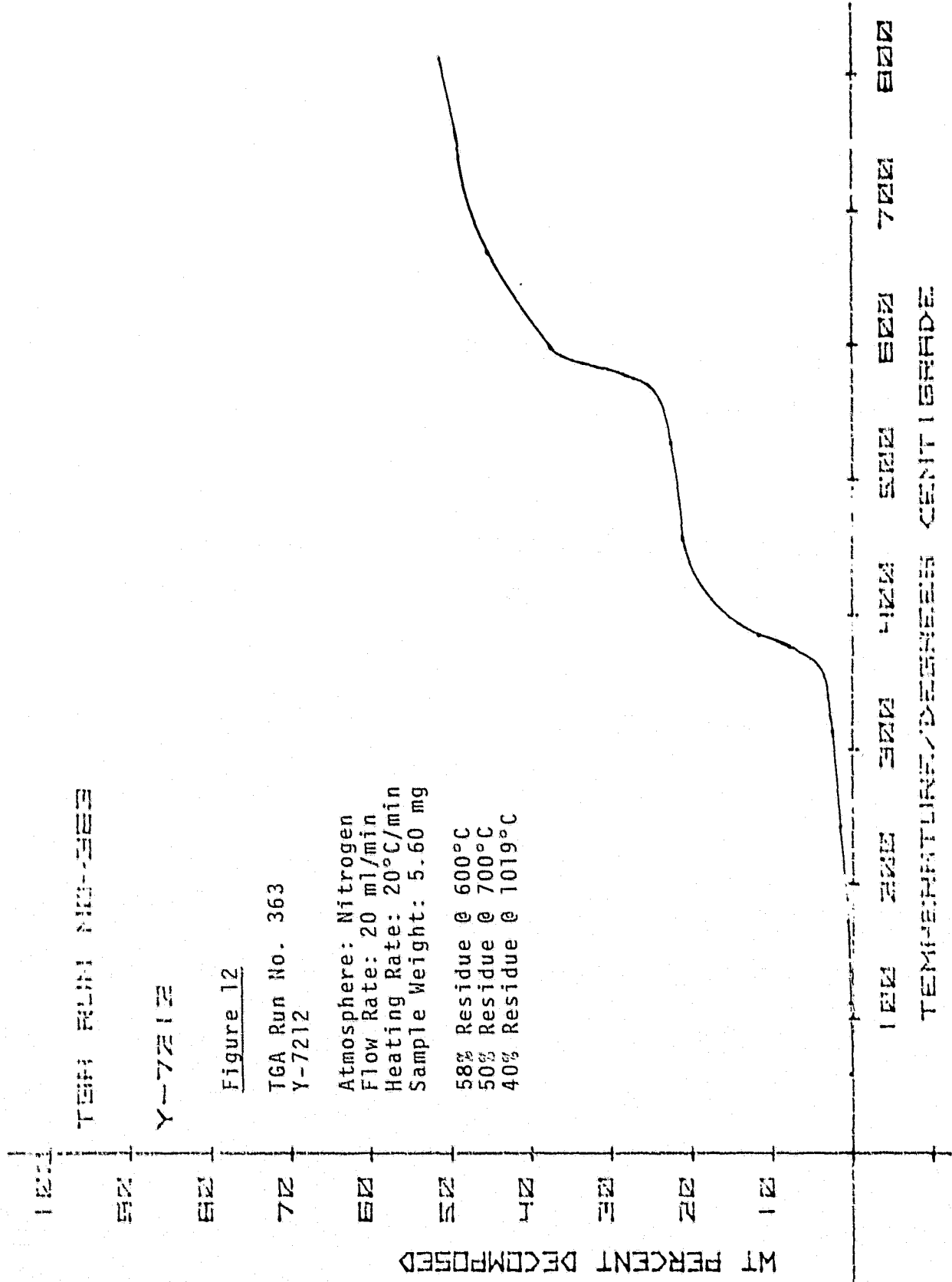
Figure 11

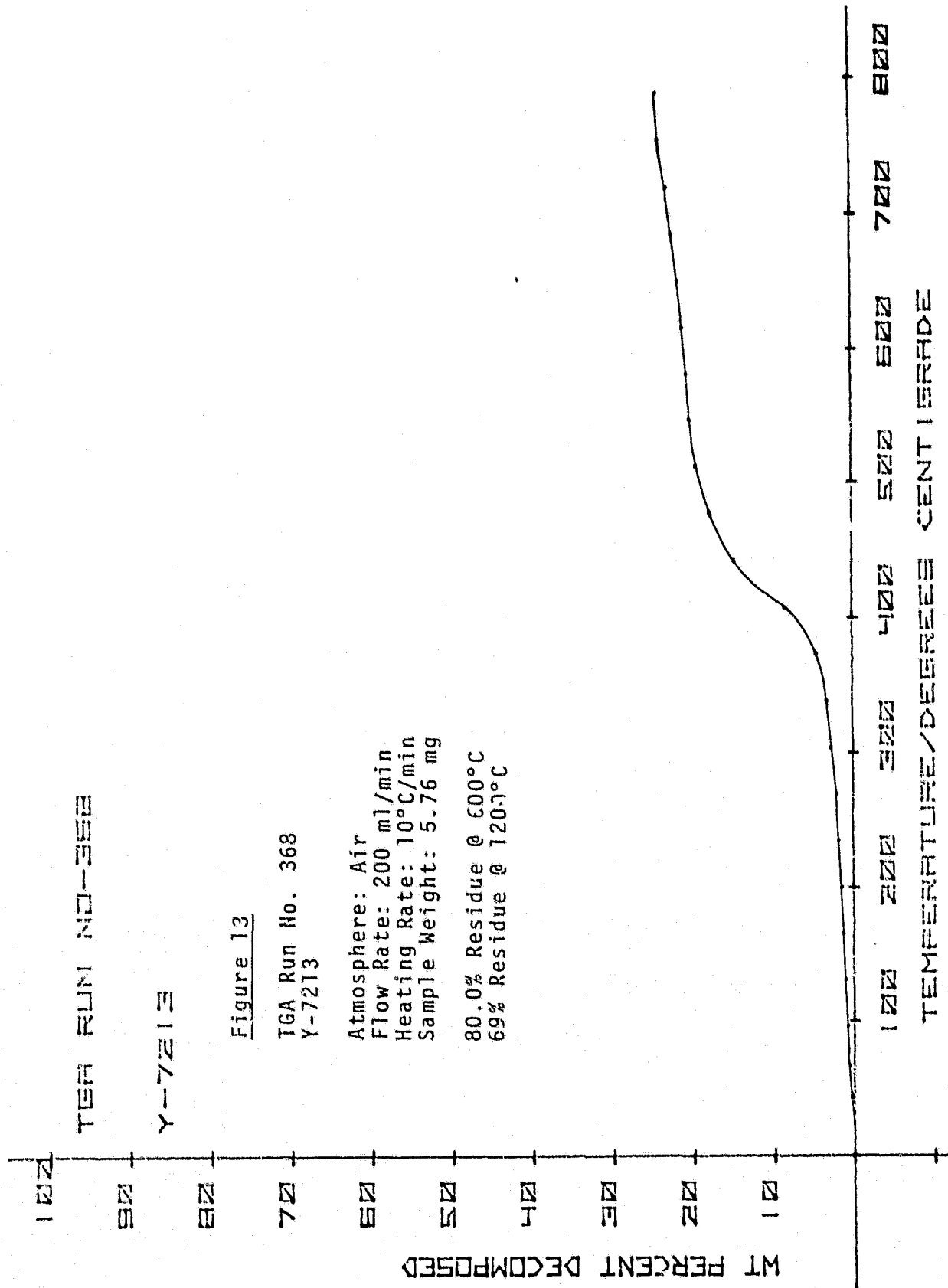
TGA Run No. 373
Y-7212

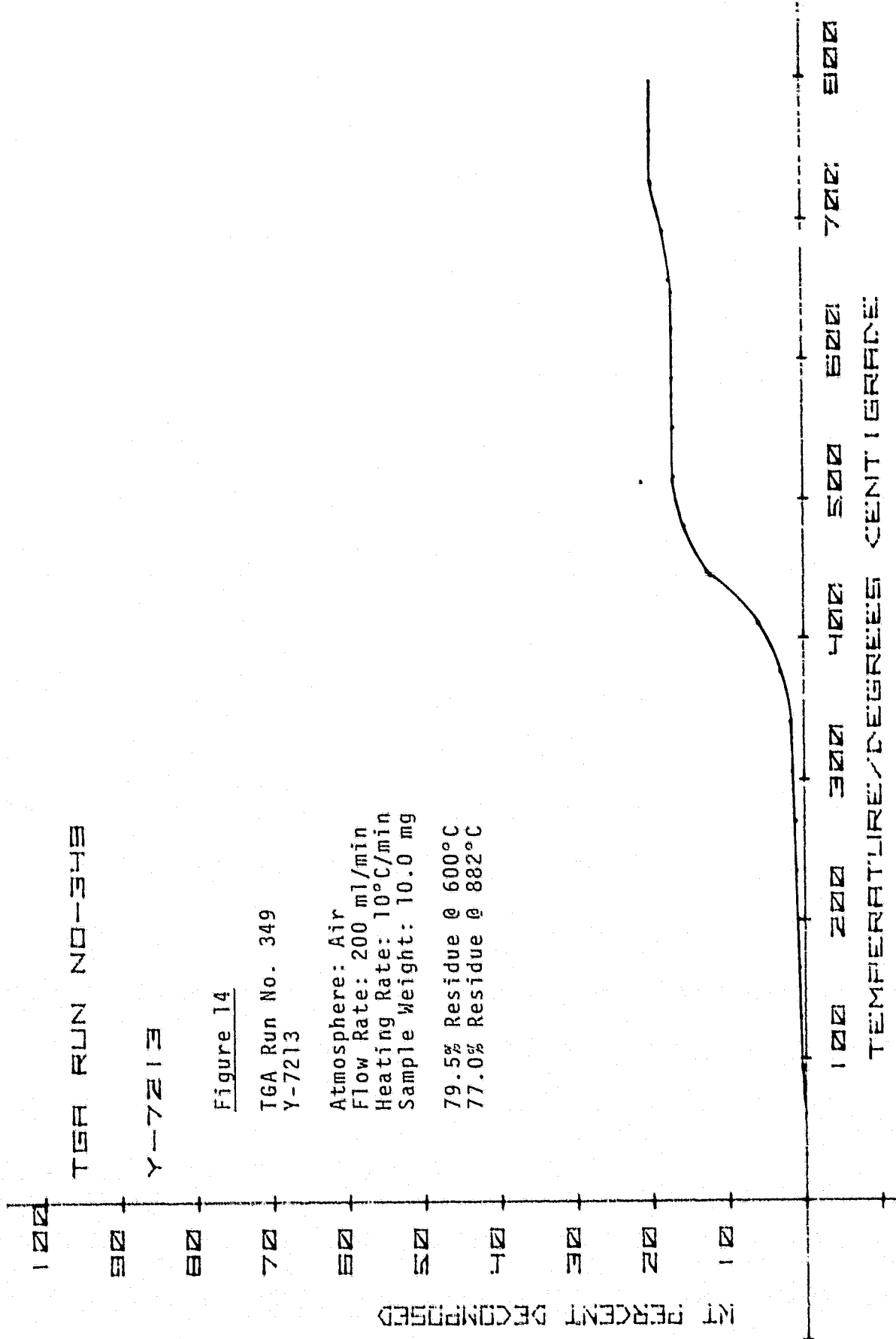
Atmosphere: Air
Flow Rate: 20 ml/min
Heating Rate: 20°C/min
Sample Weight: 7.45 mg

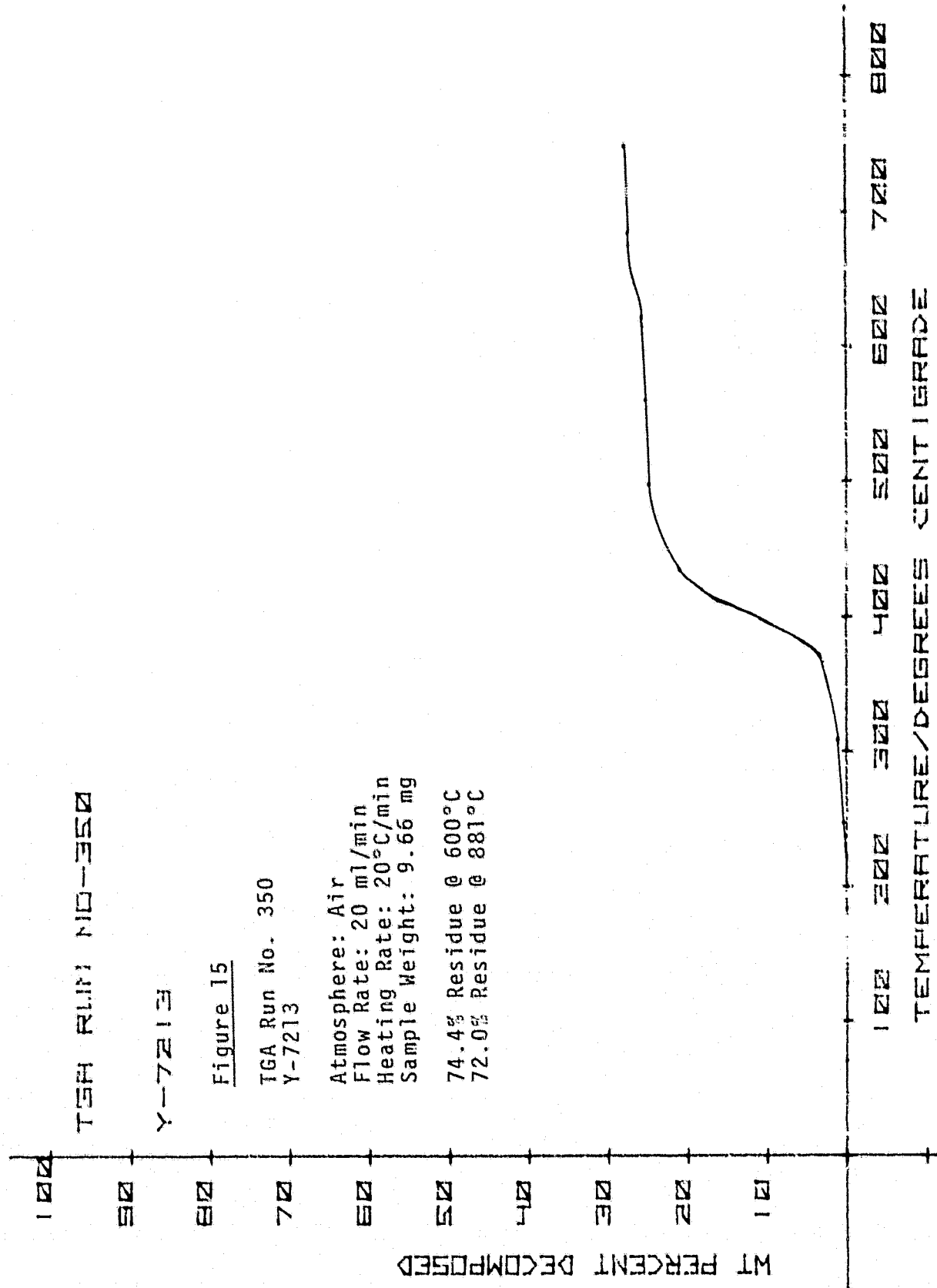
46% Residue @ 600°C
50% Residue @ 585°C
20% Residue @ 971°C

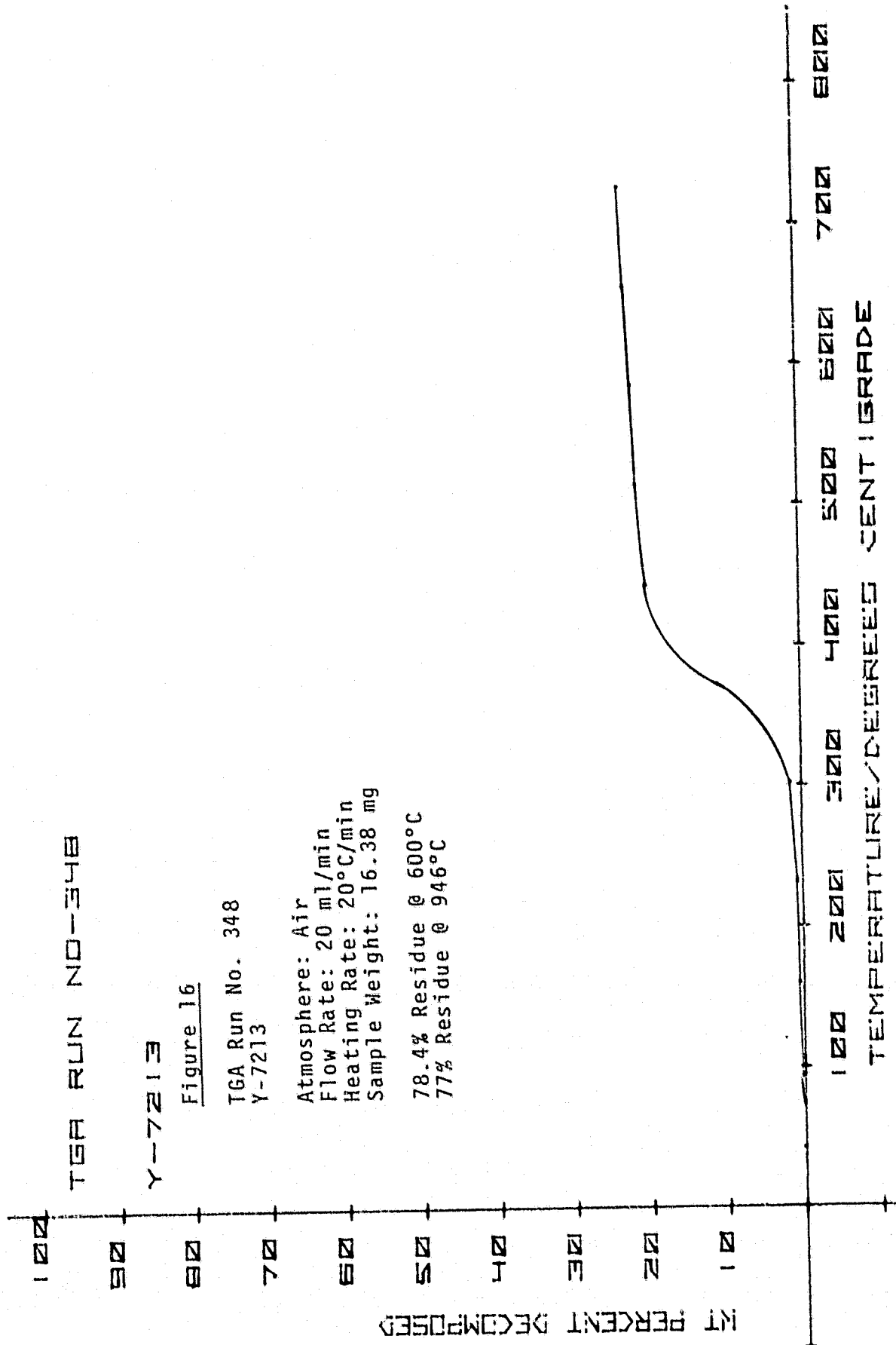


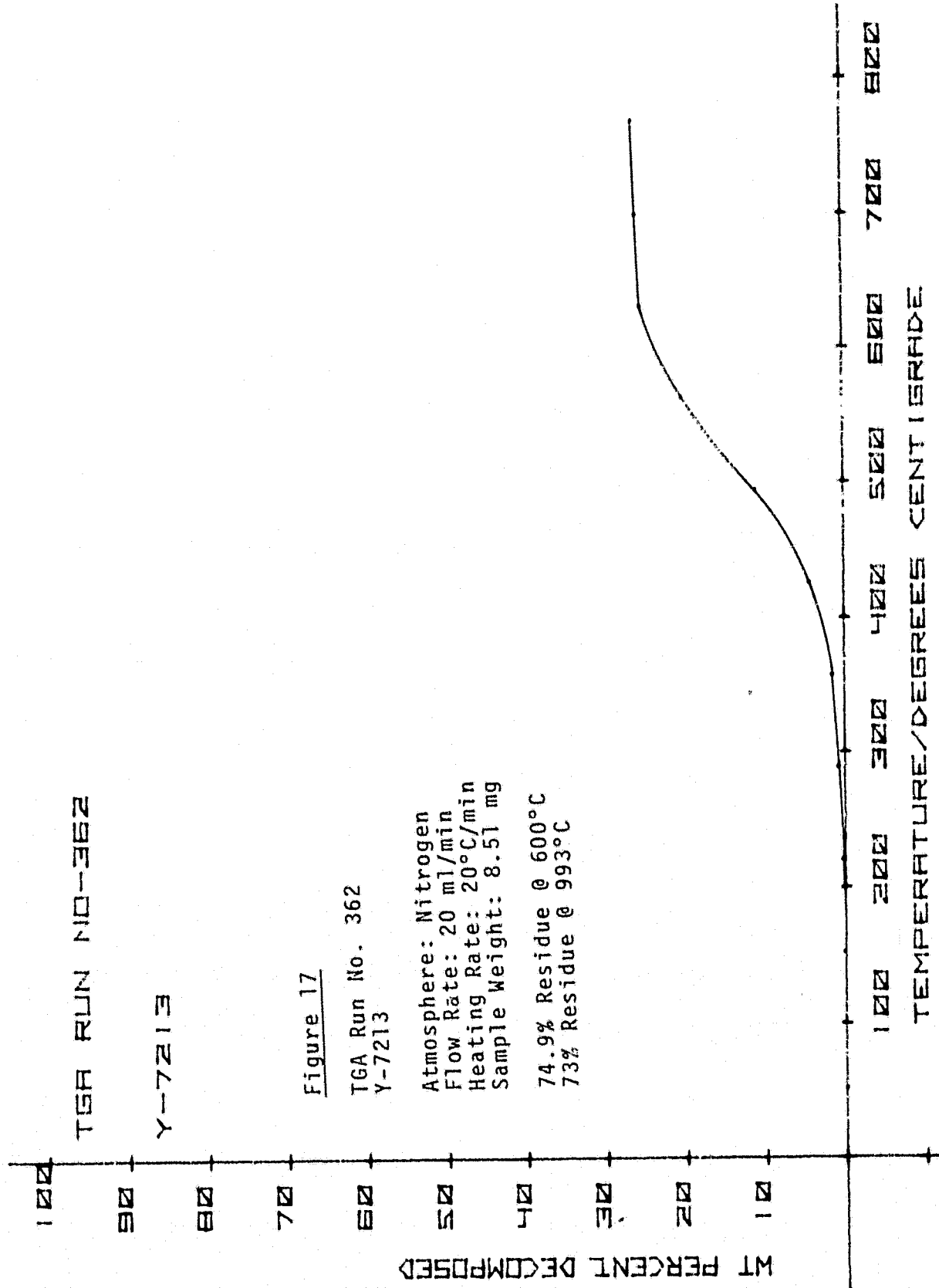


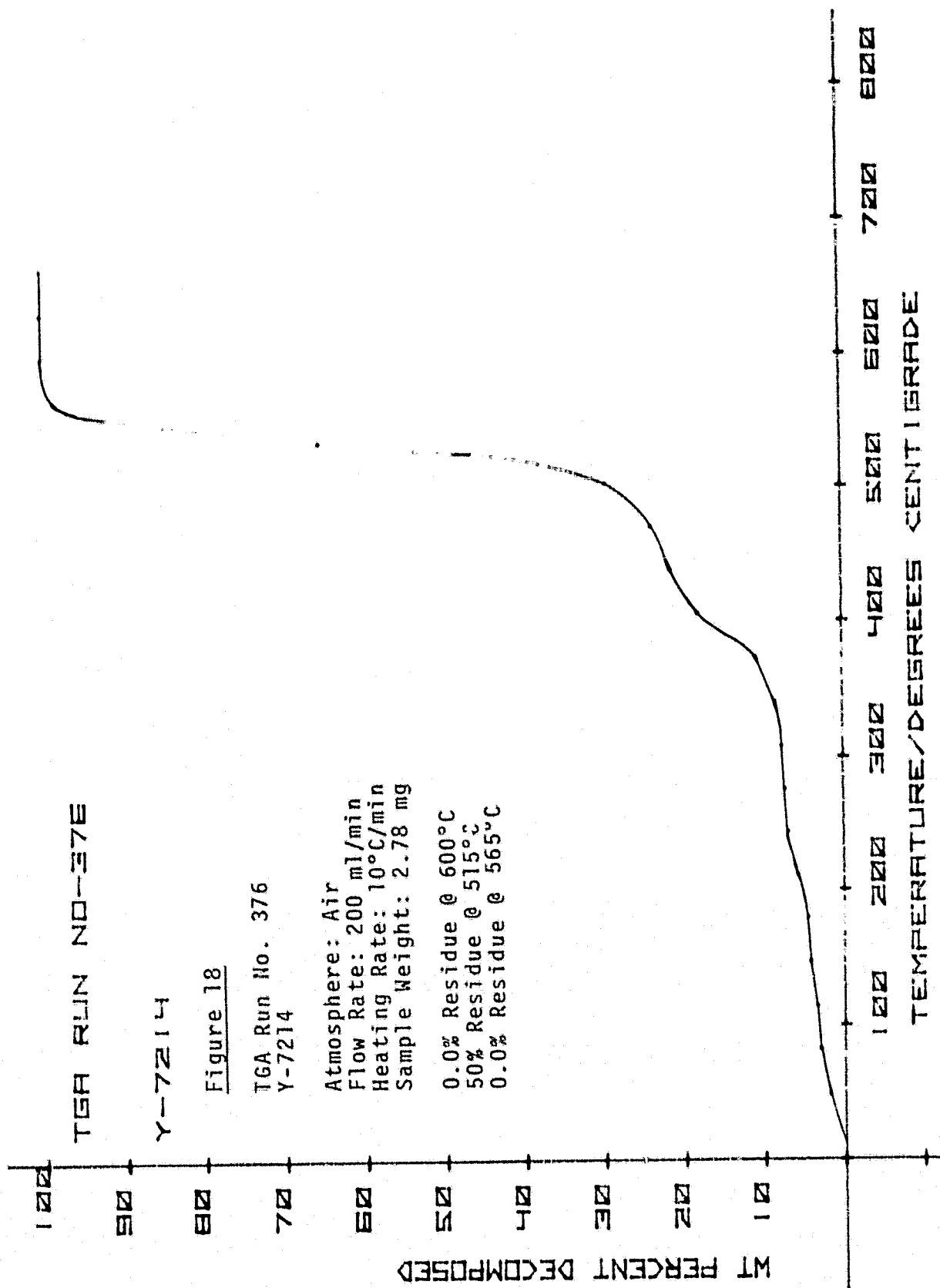


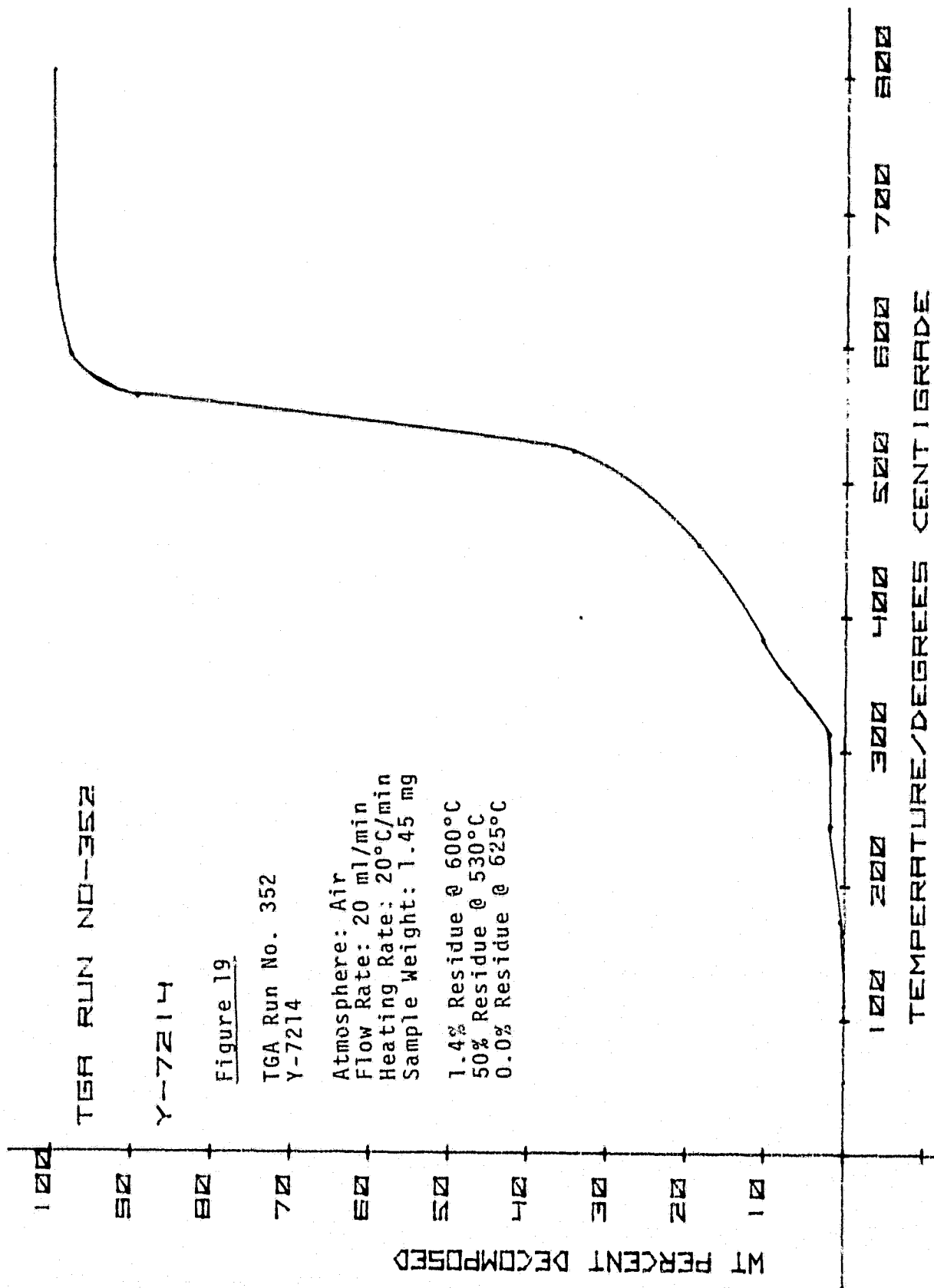


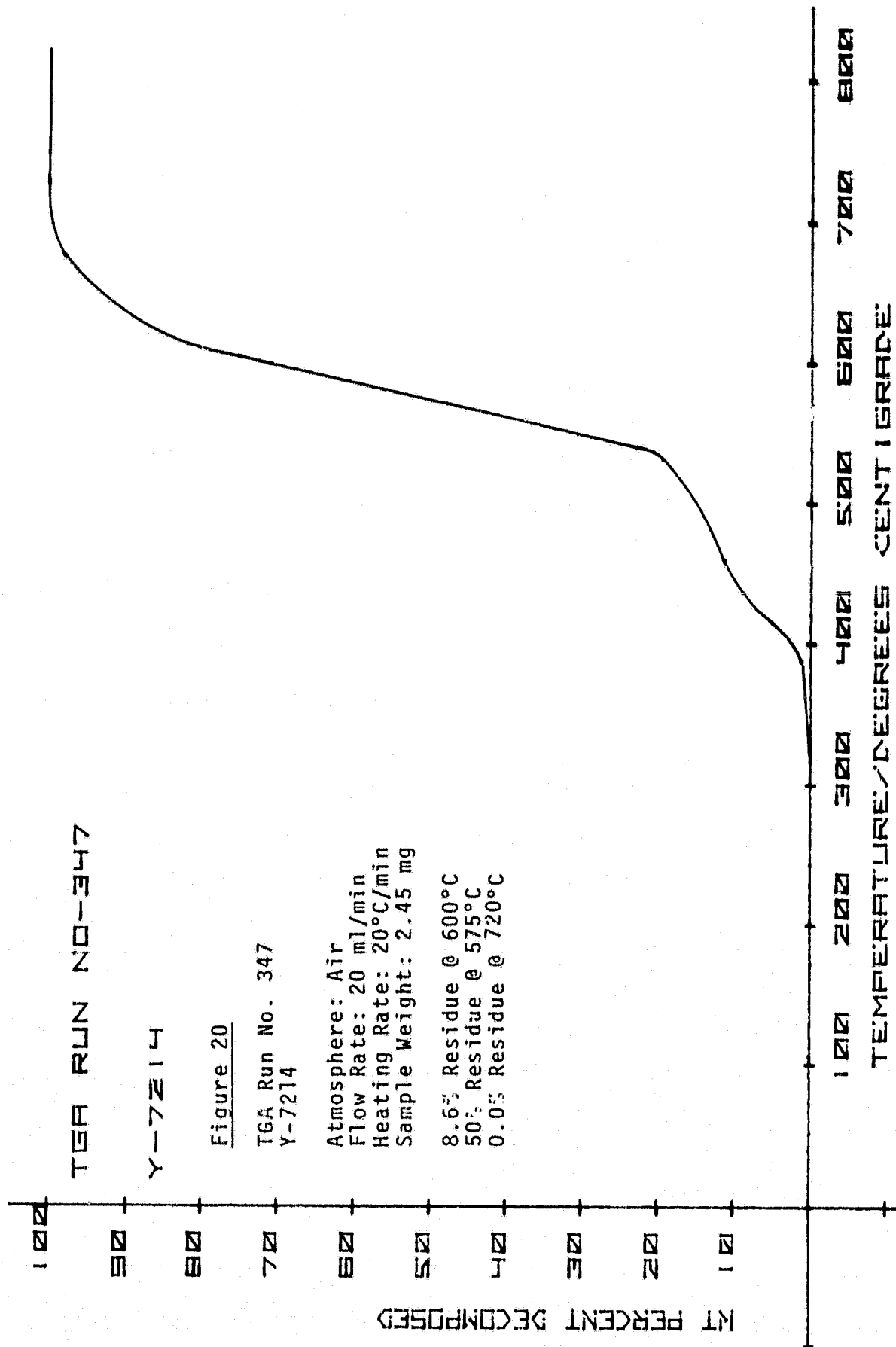












TGA RUN NO-361

Y-7214

Figure 21

TGA Run No. 361
Y-7214

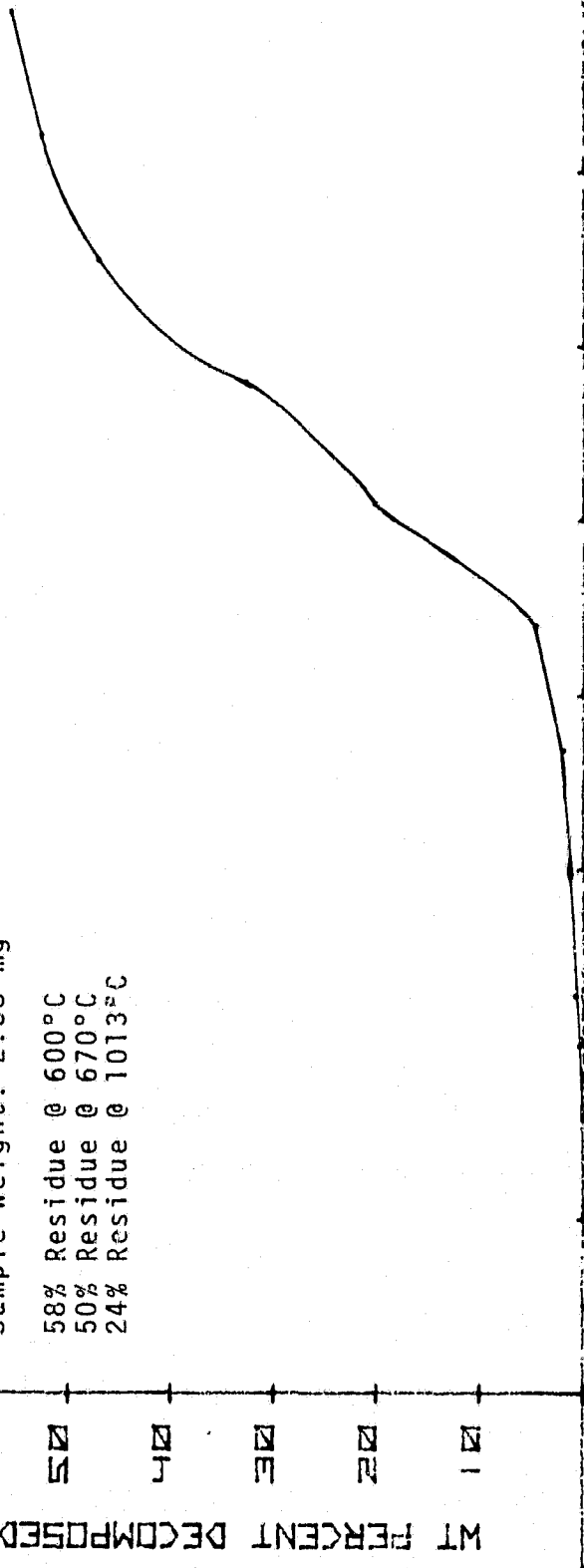
Atmosphere: Nitrogen
Flow Rate: 20 ml/min
Heating Rate: 20°C/min
Sample Weight: 2.38 mg

58% Residue @ 600°C
50% Residue @ 670°C
24% Residue @ 1013°C

WT PERCENT DECOMPOSED

100
90
80
70
60
50
40
30
20
10

100 200 300 400 500 600 700 800
TEMPERATURE/DEGREES CENTIGRADE



SHOCK-AVOIDANCE BEHAVIORAL RESPONSES TO PYROLYSATES

Figures 23 through 51 graphically depict the shock-avoidance (behavioral) response of individual rats to inhalation exposure of the designated samples. Each rat received a single pyrolysate exposure. These data are referred to as "Day 3" in the data analysis. Figure 22 is a "heat only" experiment conducted in the same manner.

The average percent shock-avoidance is plotted vs. time. Thus, the average performance of the rat in a time interval is graphed at the mid-point for that time interval, i.e., the average response for 0 to 10 minutes is plotted at 5 minutes; the average response for 60 to 65 minutes is plotted at 62.5 minutes; etc.

Shock-Avoidance Behavioral Responses to Pyrolysates

Figure 22 1) Heat only (no pyrolysate), up to 95°F (35°C)
[for reference, only]

Y-7191 Polyimide Foam (1.3 gm)

Figure 23 2) Black 2

Figure 24 3) Blue 6

Figure 25 4) Red 5

Figure 26 5) Red 12

Y-7192 Polyurethane Foam (3.4 gm)

Figure 27 6) Blue 9

Figure 28 7) Black 6

Figure 29 8) Black 8

Figure 30 9) Red 13

Y-7212 Orcon Kapton with Kel-F (2.0 gm)

Figure 31 10) Black 1

Figure 32 11) Red 3

Figure 33 12) Red 6

Figure 34 13) Red 22

Y-7213 3M Silicone on Fiberglass (18.5 gm)

Figure 35 14) Red 7

Figure 36 15) Red 8

Figure 37 16) Red 9

Figure 38 17) Red 20

Y-7214 Polyimide Foam, #720-1 (2.4 gm)

Figure 39 18) Black 3

Figure 40 19) Red 1

Figure 41 20) Red 2

Figure 42 21) Red 14

Y-7192 Polyurethane Foam (1.7 gm; approx. 1/4 LD₅₀)

- Figure 43 22) Black 9
- Figure 44 23) Red 15
- Figure 45 24) Red 17
- Figure 46 25) Red 19

Additional Shock-Avoidance Behavioral Patterns to Pyrolysates
Using Other Quantities of Samples for Response Trends, but Which
Were Not Included in the Statistical Analyses.

Y-7212 Orcon Kapton with Kel F (1.1 gm; approx.
1/4 of LD₅₀)

- Figure 47 26) Black 7
- Figure 48 27) Red 4

Y-7214 Polyimide Foam, #720-1 (3.6 gm; approx. 3/4
of LD₅₀)

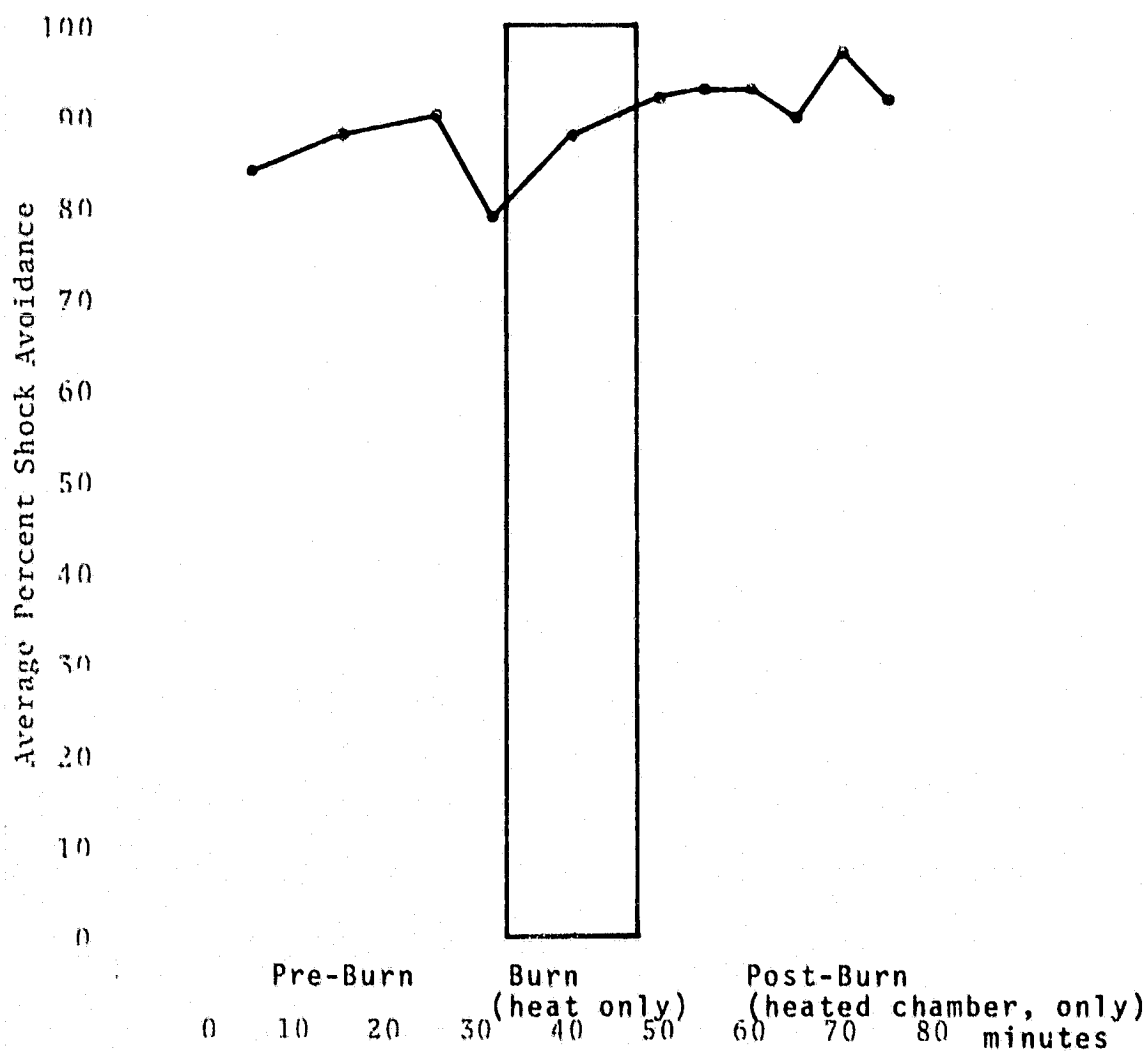
- Figure 49 28) Red 25
- Figure 50 29) Red 26
- Figure 51 30) Red 27

Figure 22

Sample: None

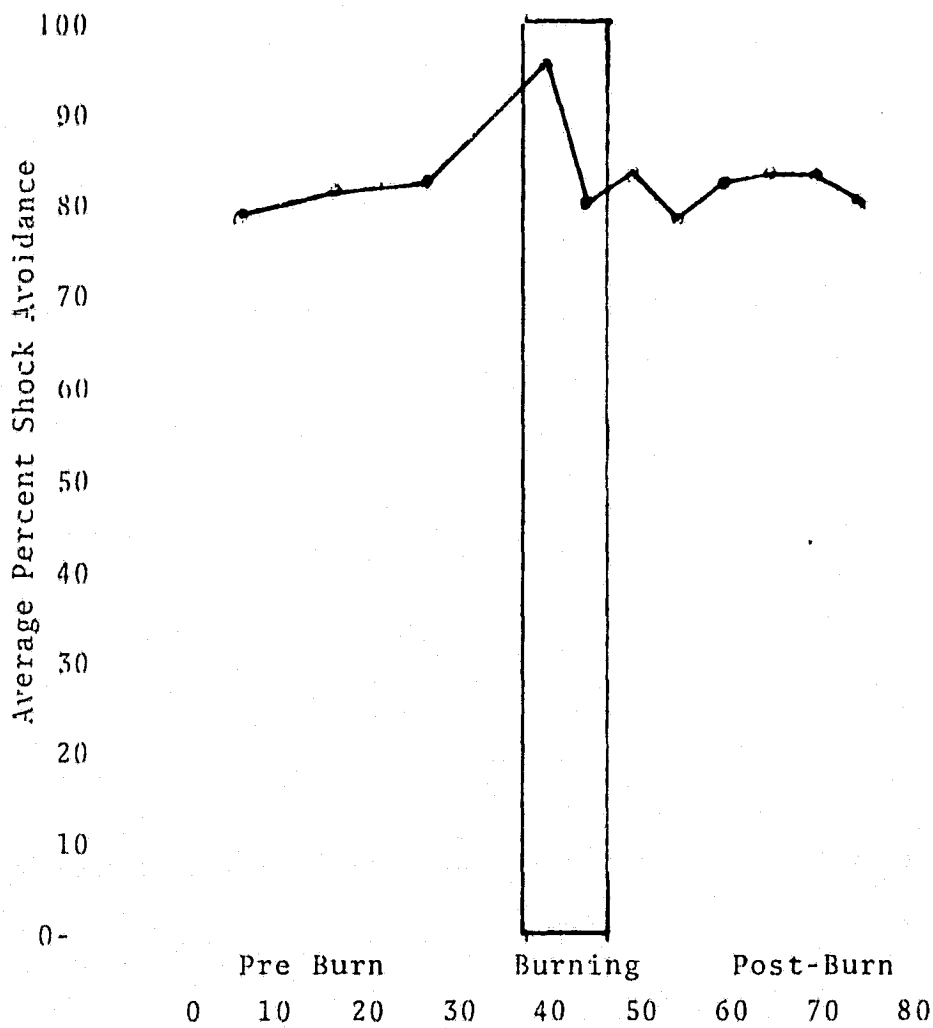
Test conducted to determine rat's response
to heat only [produced by pyrolysis/combustion
furnace used for thermodegradation of samples]

Red 26



Maximum Chamber Temperature: 95°F (35°C).

Figure 23
Sample-Y-7191
Black 2

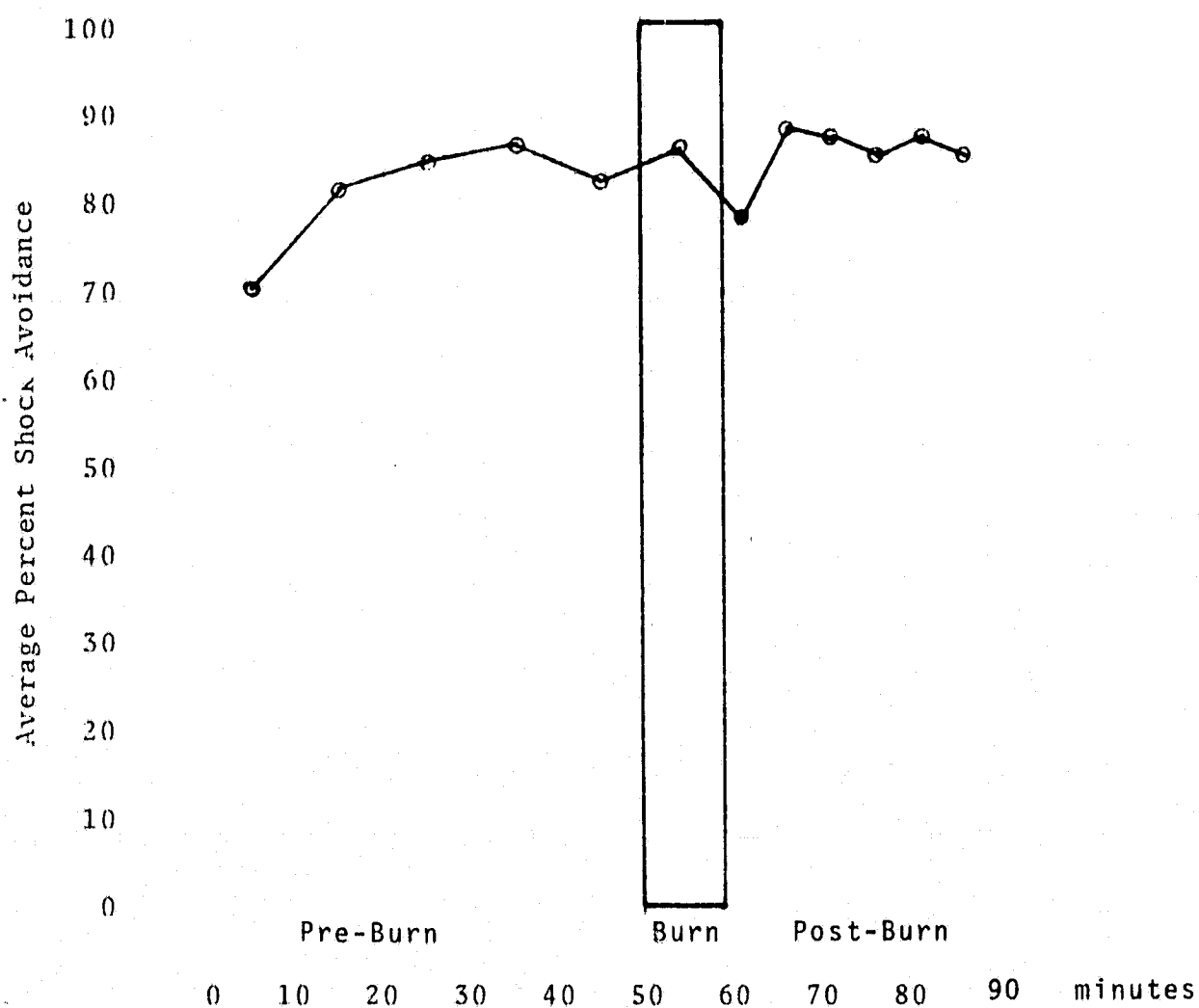


Sample Weight: 1.3 gm.

Maximum Chamber Temperature: 90°F

Figure 24

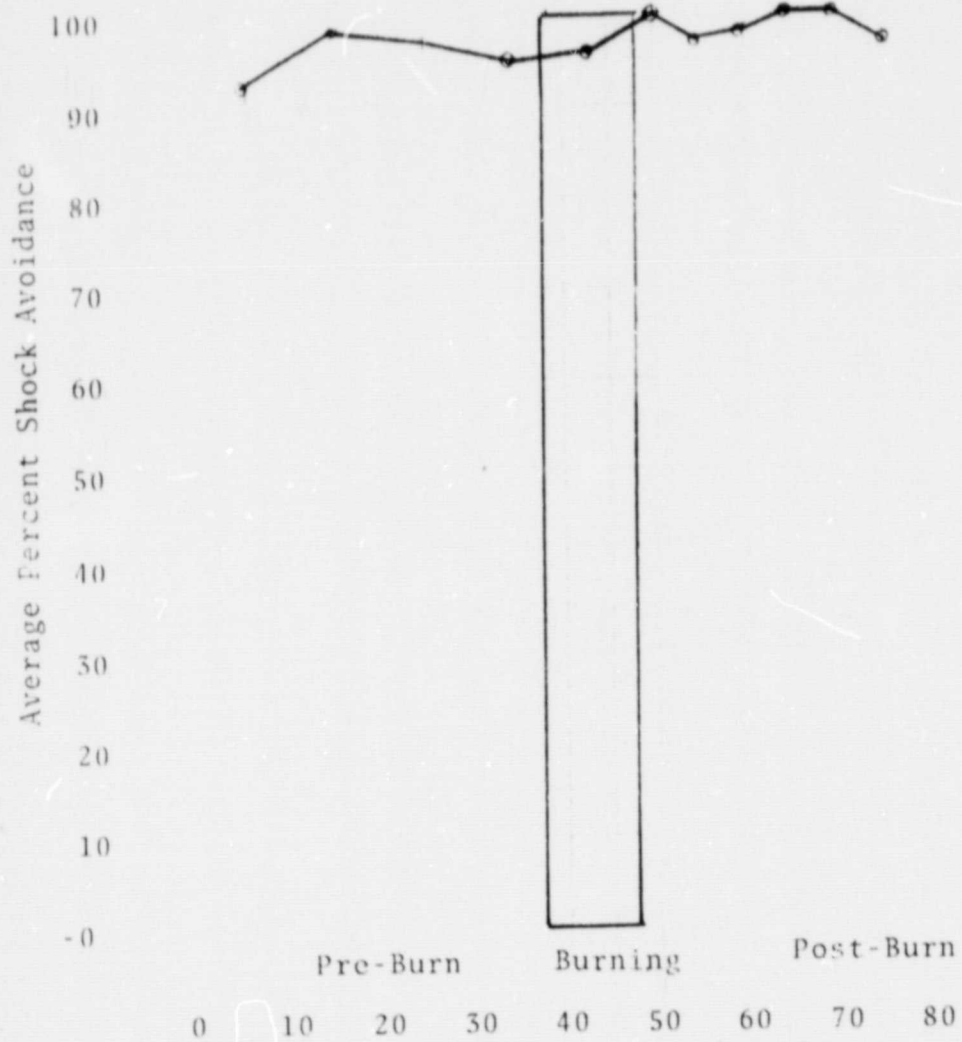
Sample Y-7191
Blue 6



Sample Weight: 1.3 gm

Maximum Chamber Temperature: 84°F.

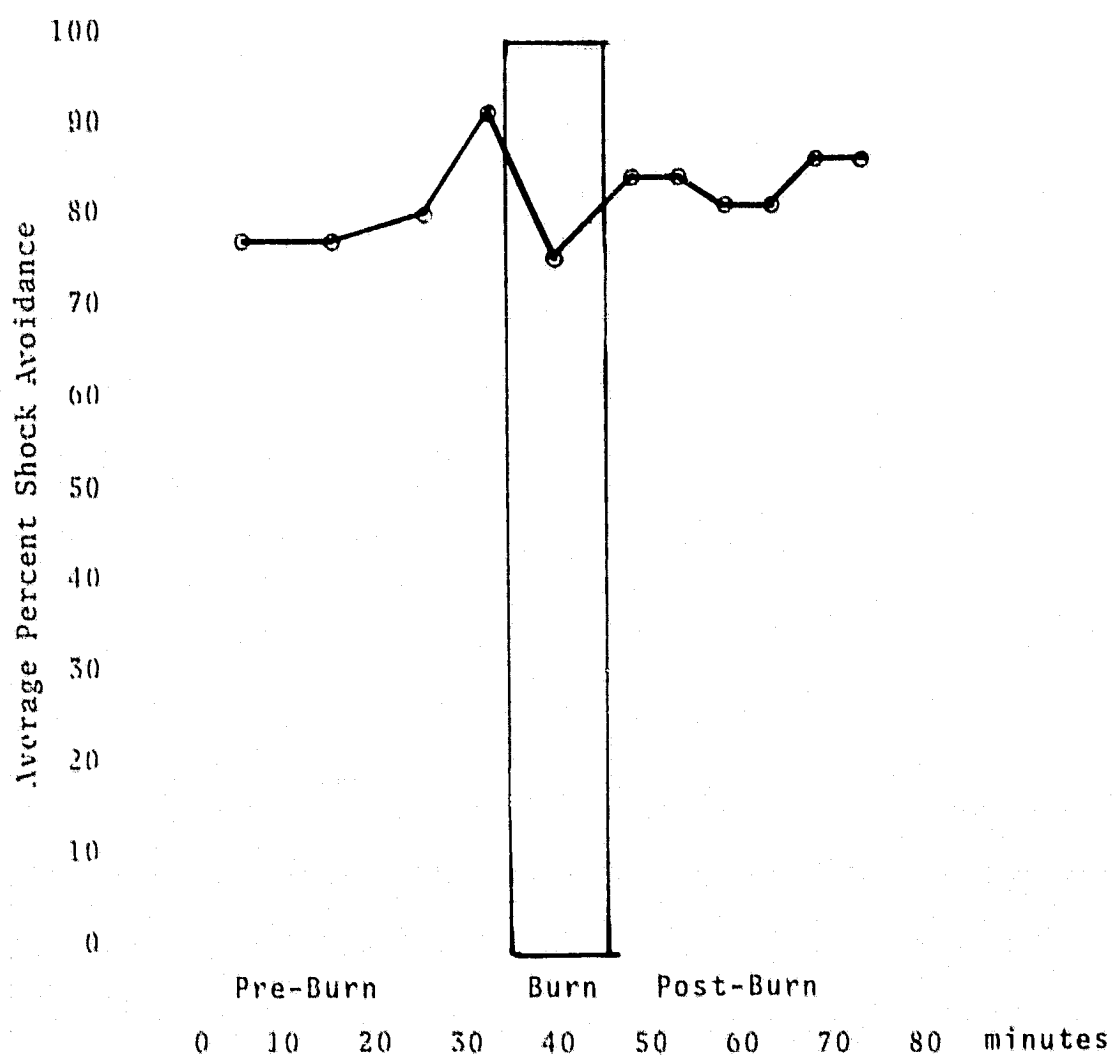
Figure 25
Sample Y-7191
Red 5



Sample Weight: 1.3 gm
Maximum Chamber Temperature: 88°F.

Figure 26

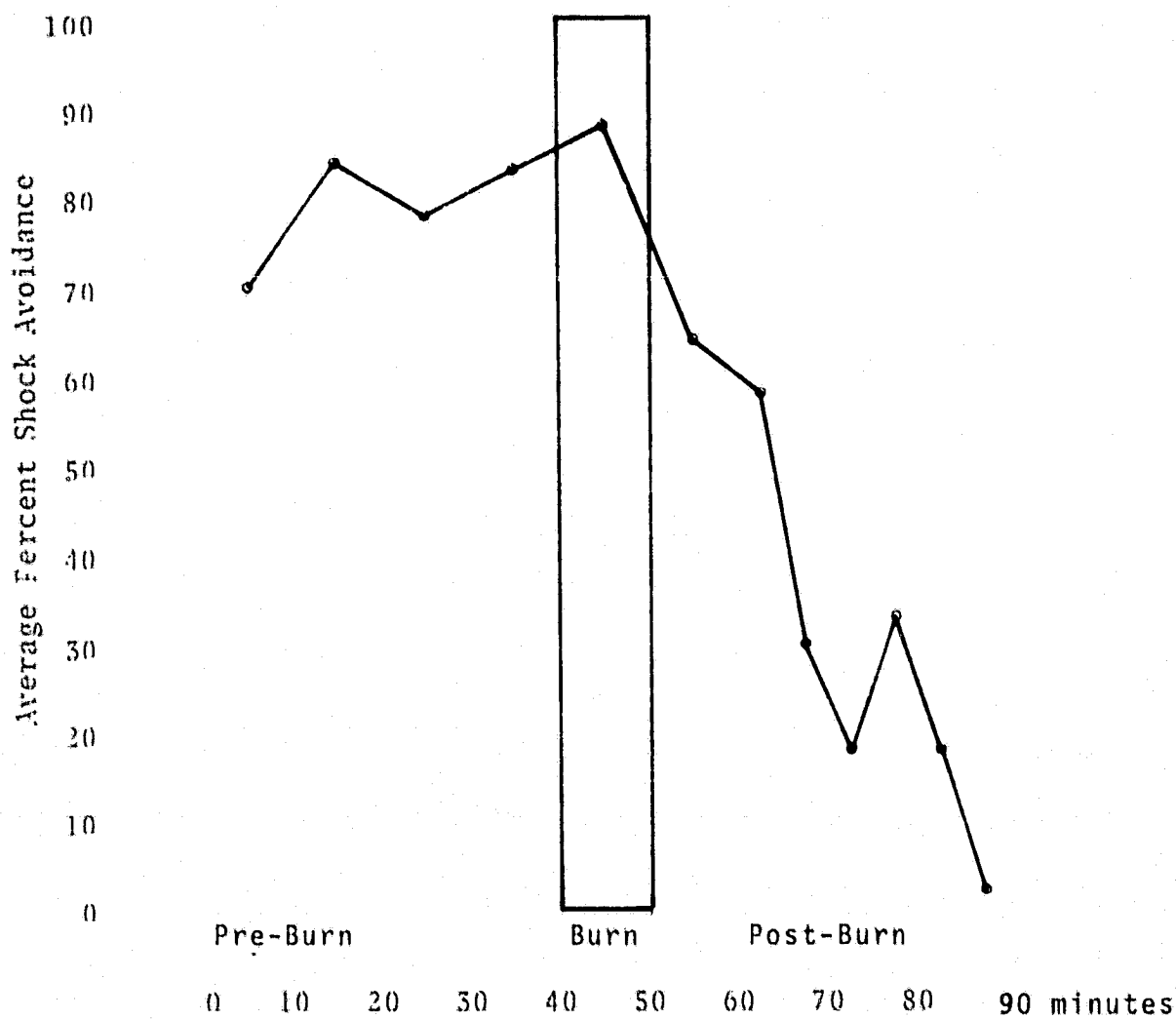
Sample Y-7191
Red 12



Sample Weight: 1.3 gm.
Maximum Chamber Temperature: 86°F

Figure 27

Sample Y-7192
Blue 9



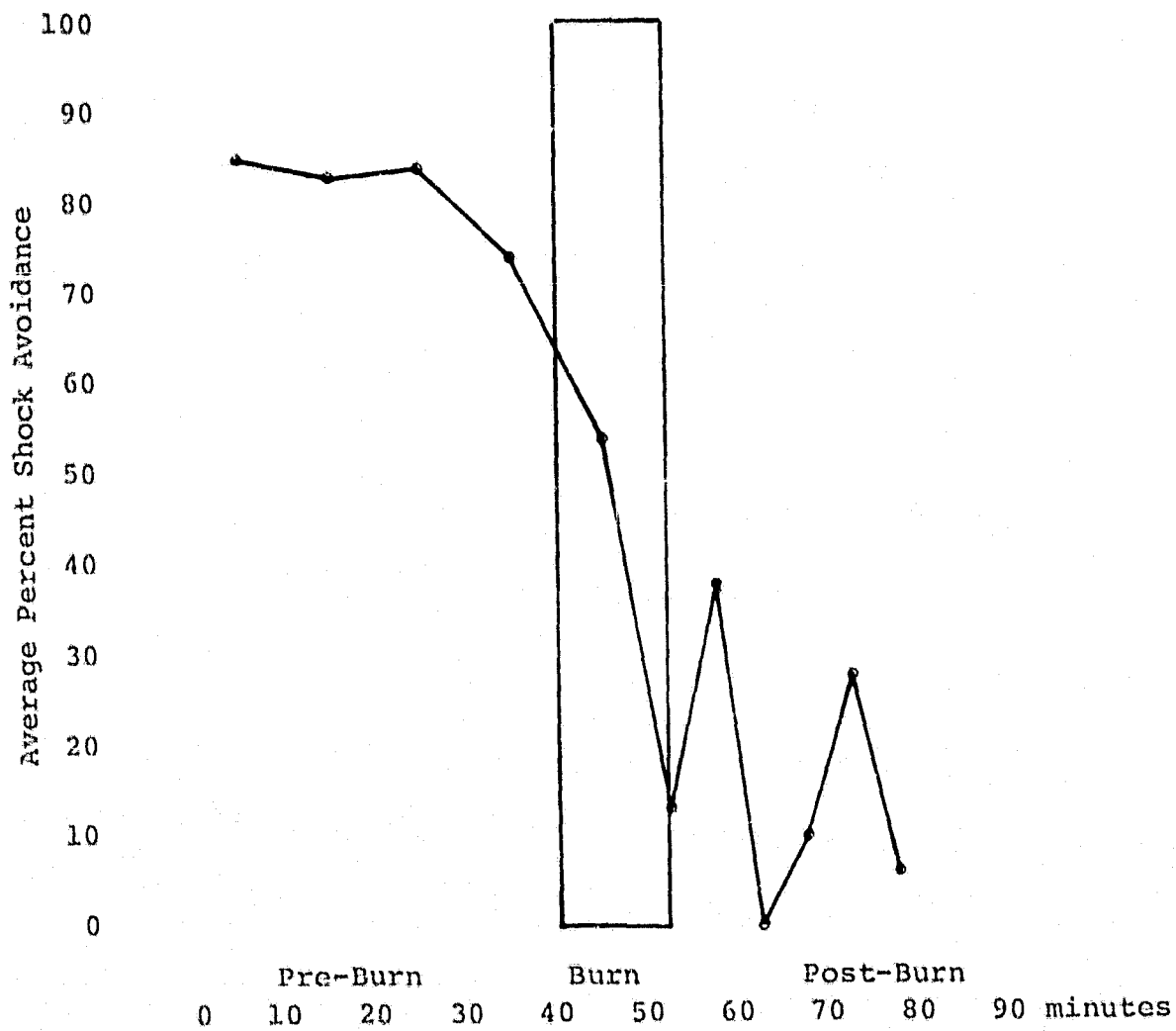
Sample Weight: 3.4 gm.

Maximum Chamber Temperature: 88°F.

Figure 28

Sample Y-7192

Black 6



Sample Weight: 3.4 gm.

Maximum Chamber Temperature: 88°F.

Figure 29

Sample Y-7192

Black 8

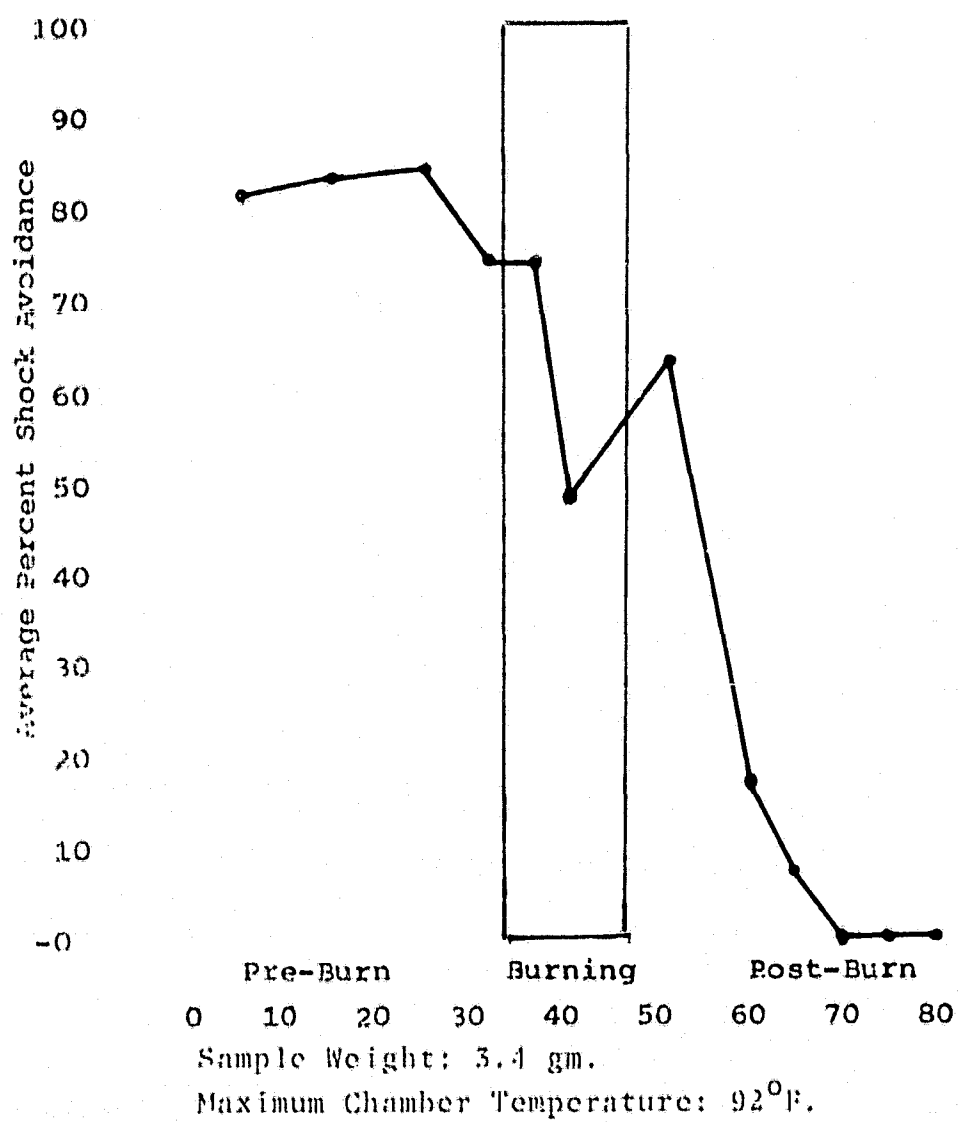
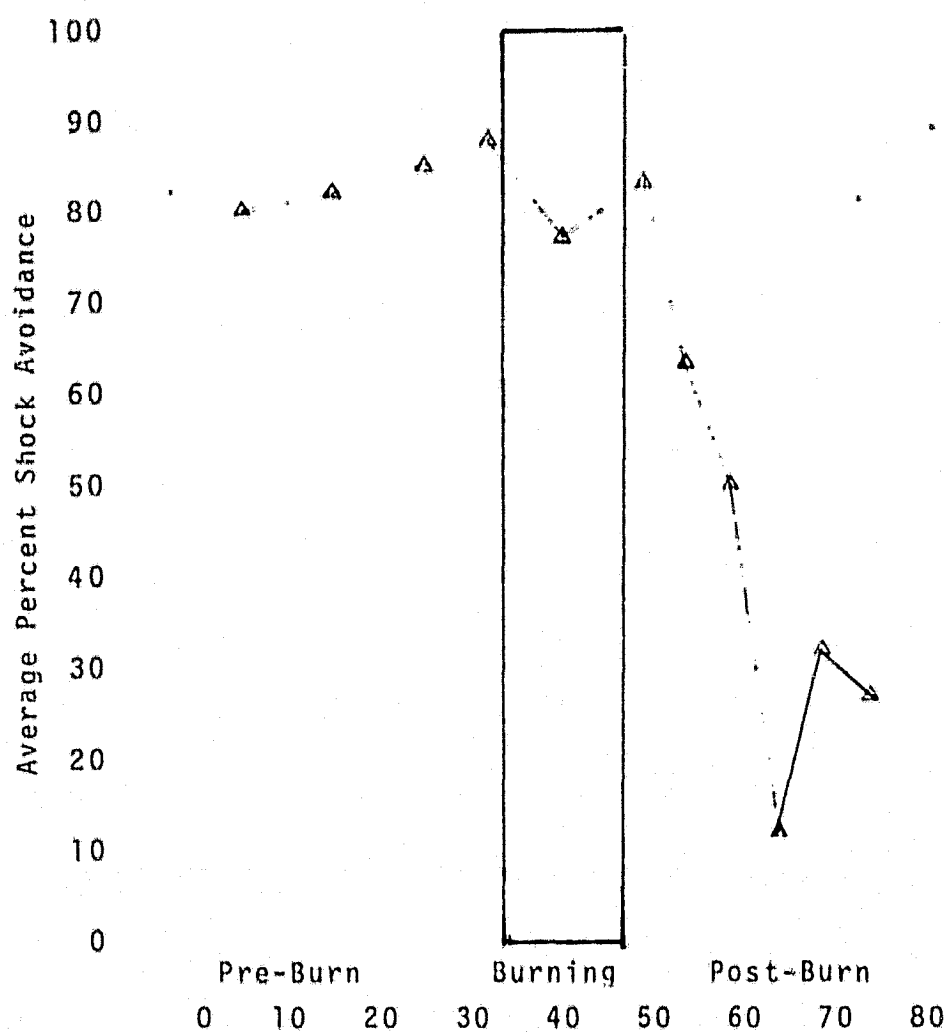


Figure 30

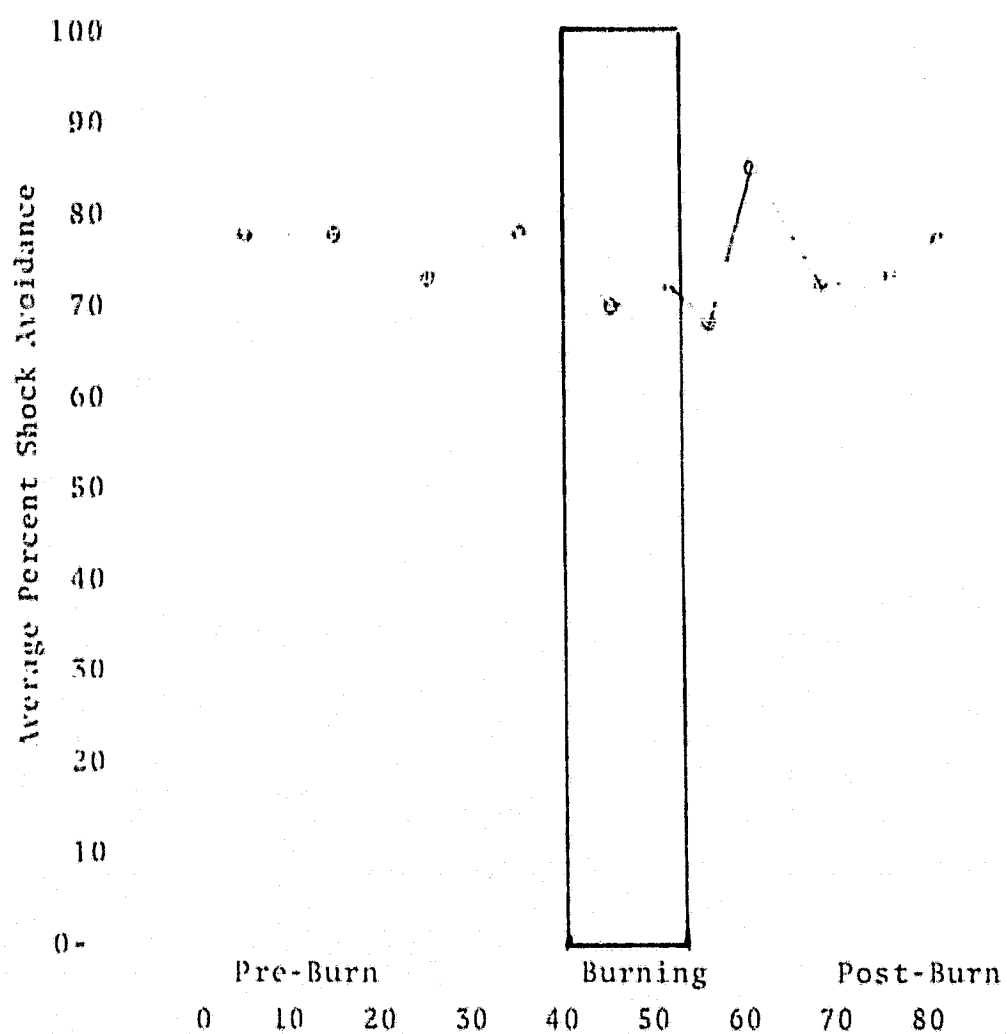
Sample Y-7192
Red 13



Sample Weight: 3.4 gm.
Maximum Chamber Temperature: 90°F.

Figure 31

Sample-Y-7212
Black-1

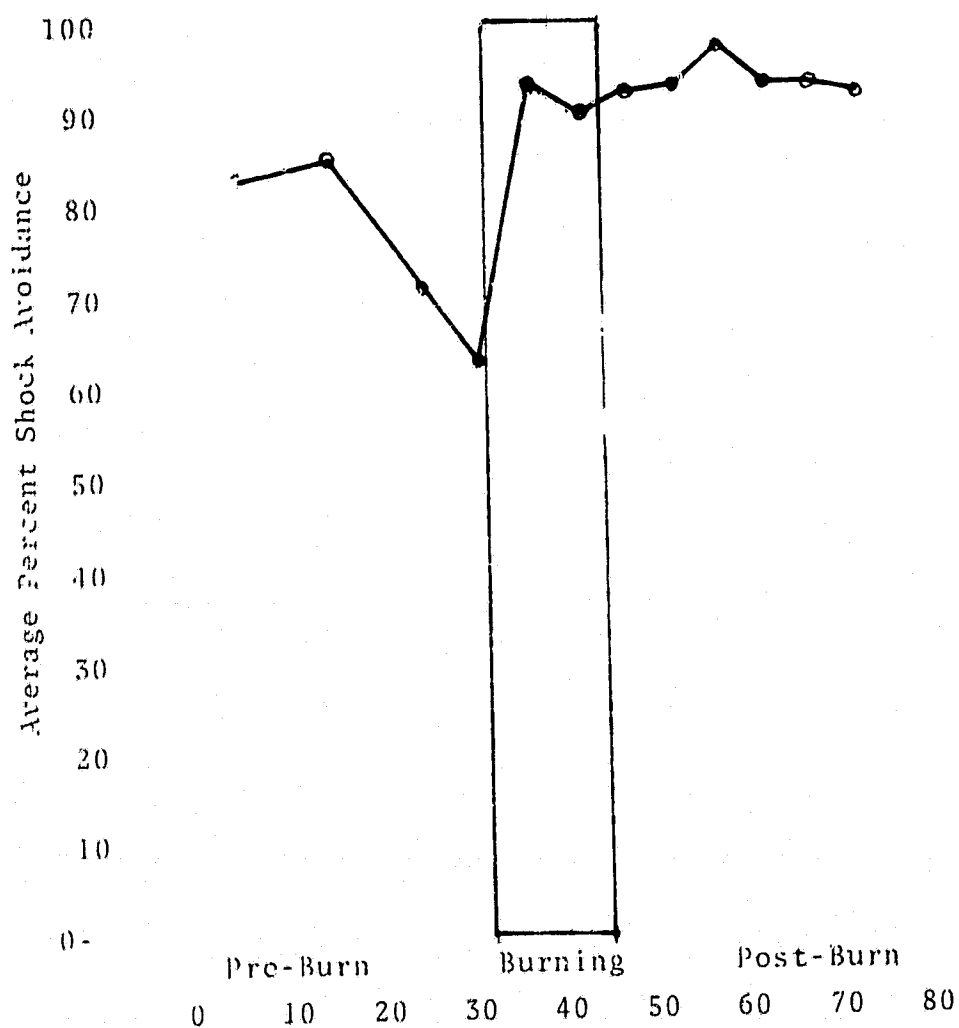


Sample Weight: 2.0 gm.

Maximum Chamber Temperature: 89°F.

Figure 32

Sample Y-7212
Red 3

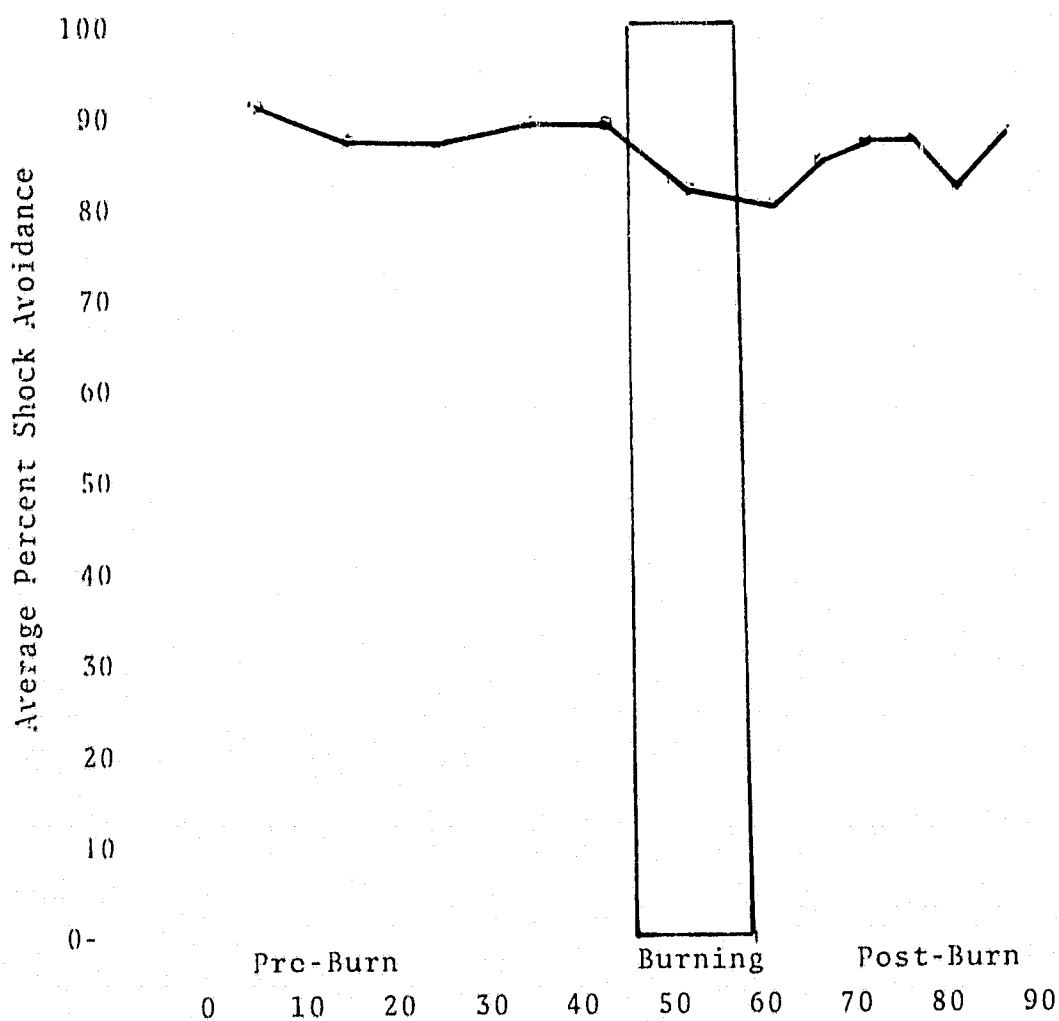


Sample Weight: 2.0 gm.

Maximum Chamber Temperature: 92°F.

Figure 33

Sample- Y-7212
Red 6



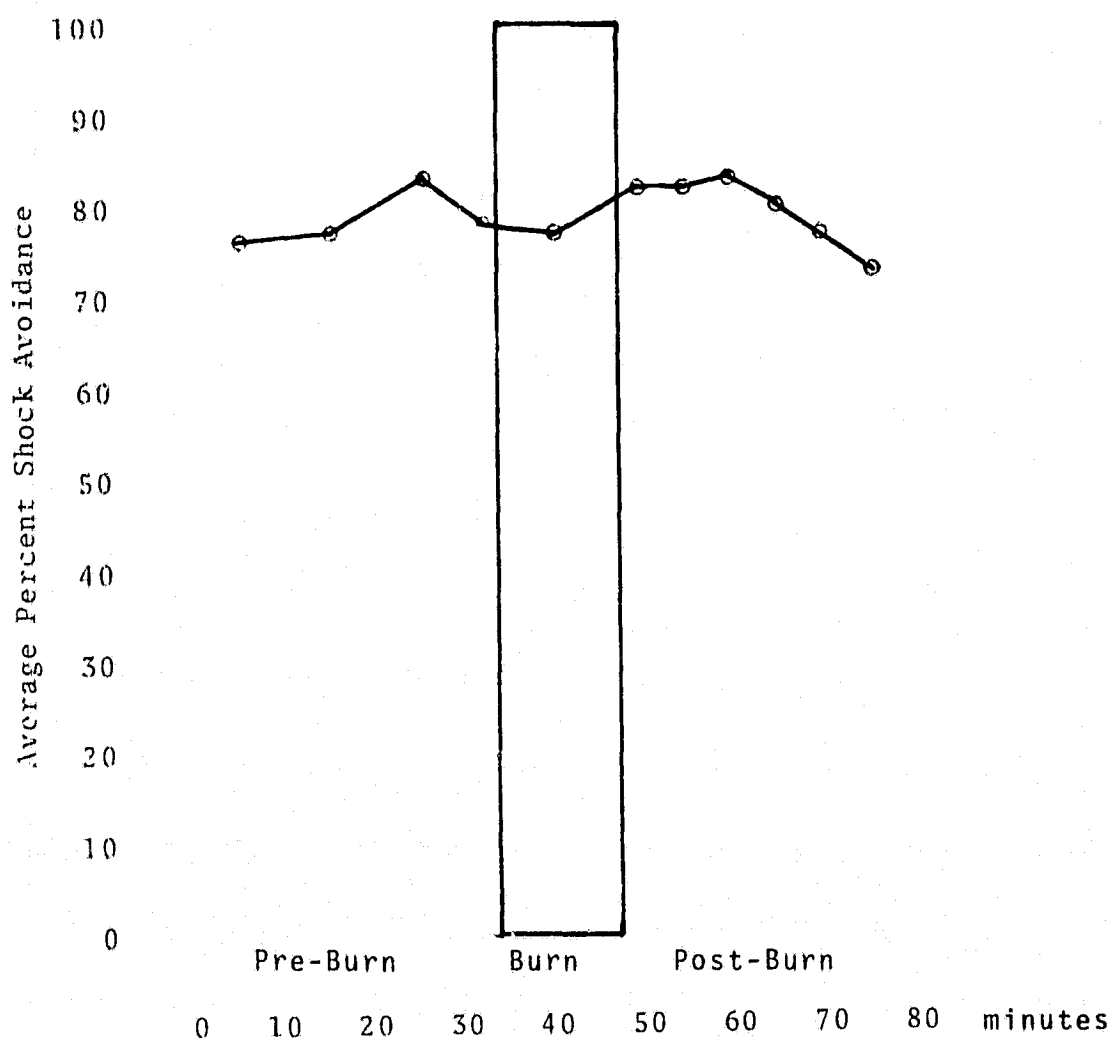
Sample Weight: 2.0 gm.

Maximum Chamber Temperature: 89°F.

Figure 34

Sample Y-7212

Req 22

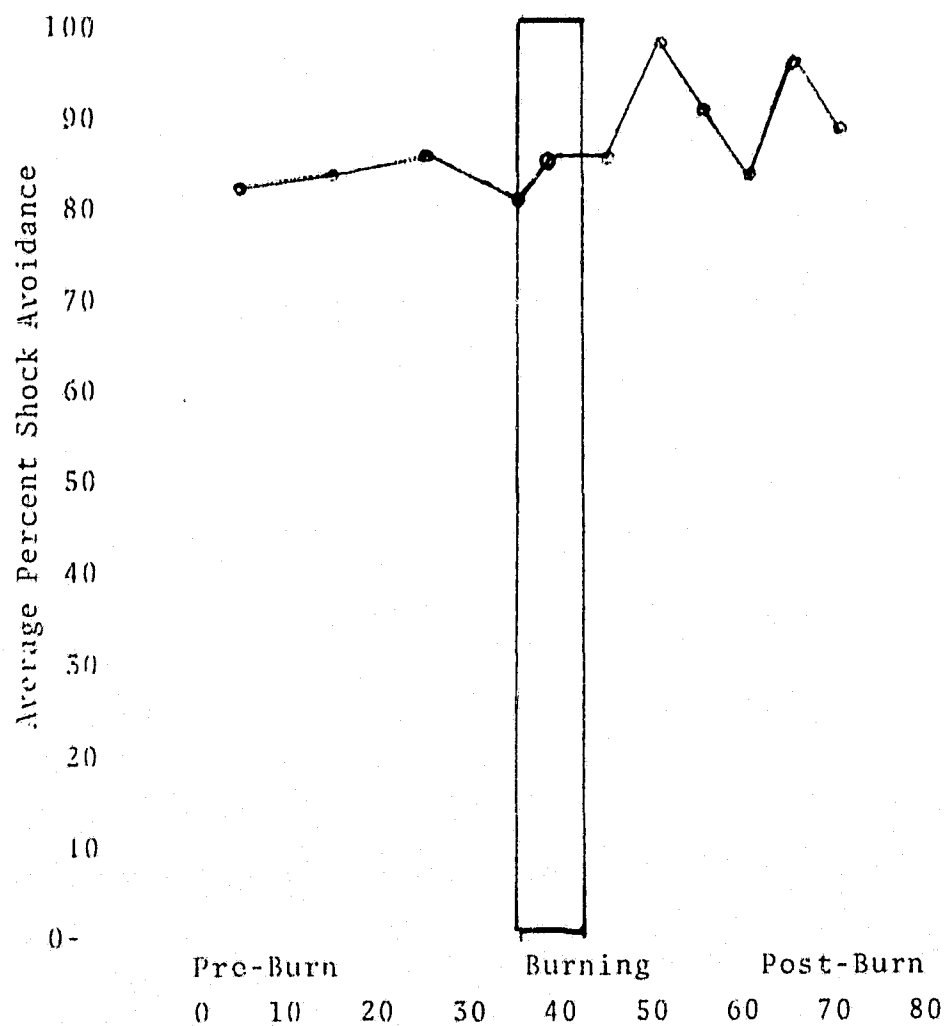


Sample Weight: 2.0 gm.

Maximum Chamber Temperature: 93°F.

Figure 35

Sample-Y-7213
Rod-7

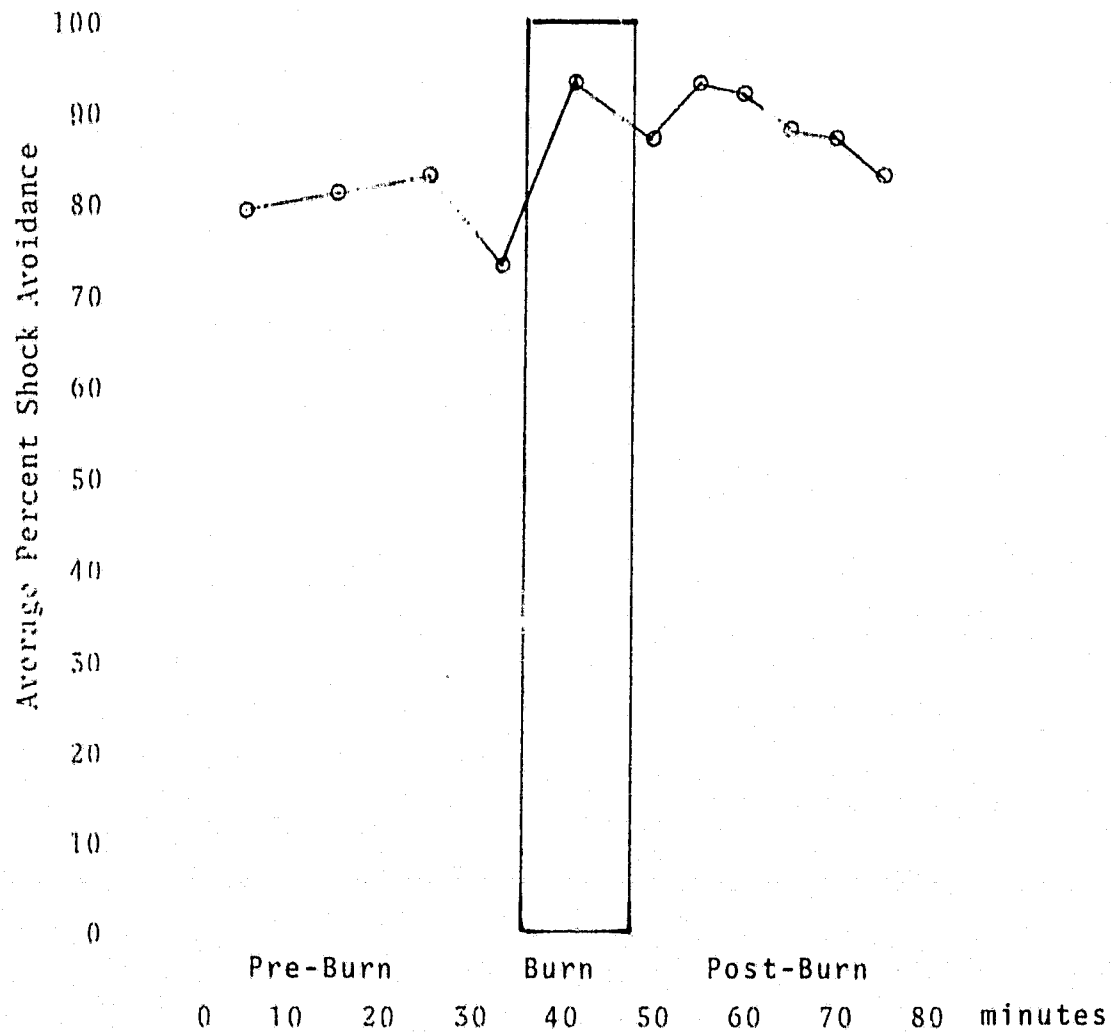


Sample Weight: 18.5 gm.

Maximum Chamber Temperature: 88°F.

Figure 36

Sample Y-7213
Red 8



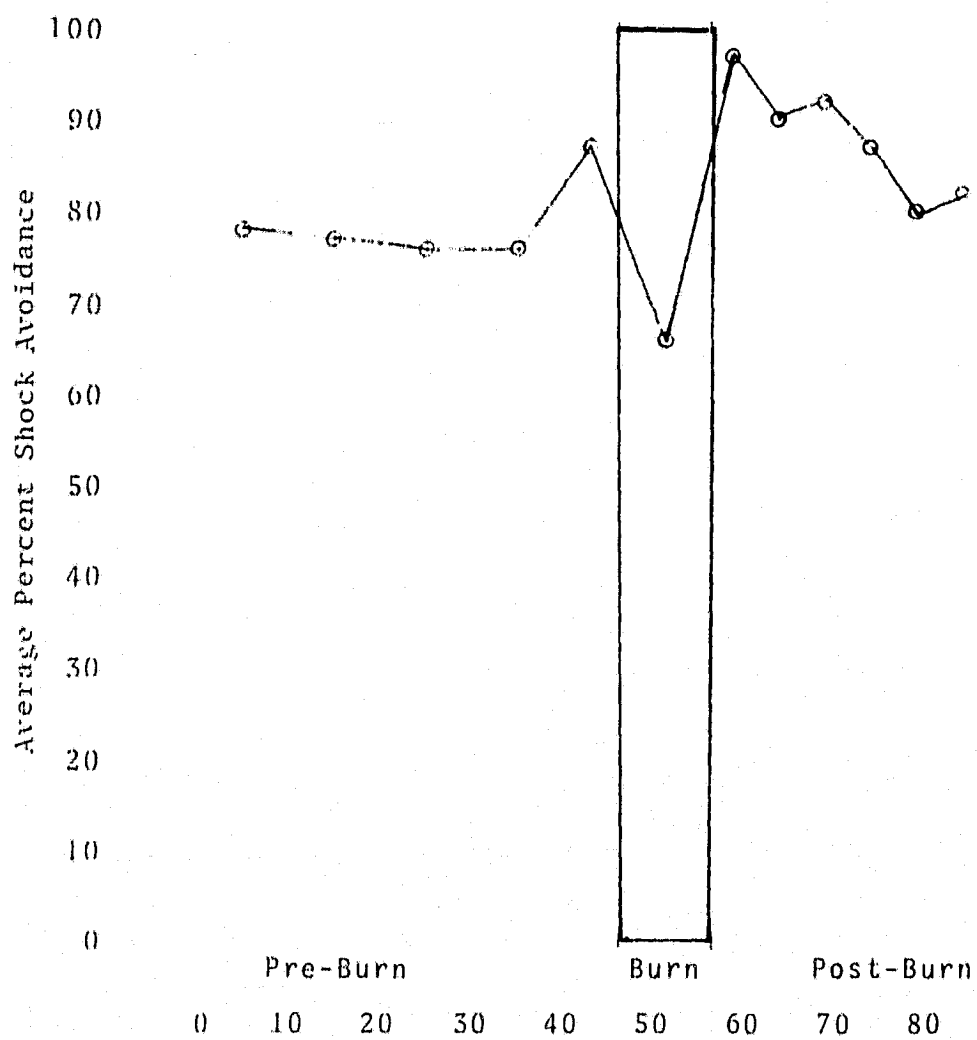
Sample Weight: 18.5 gm.

Maximum Chamber Temperature: 92°F.

Figure 37

Sample Y-7213

Red 9

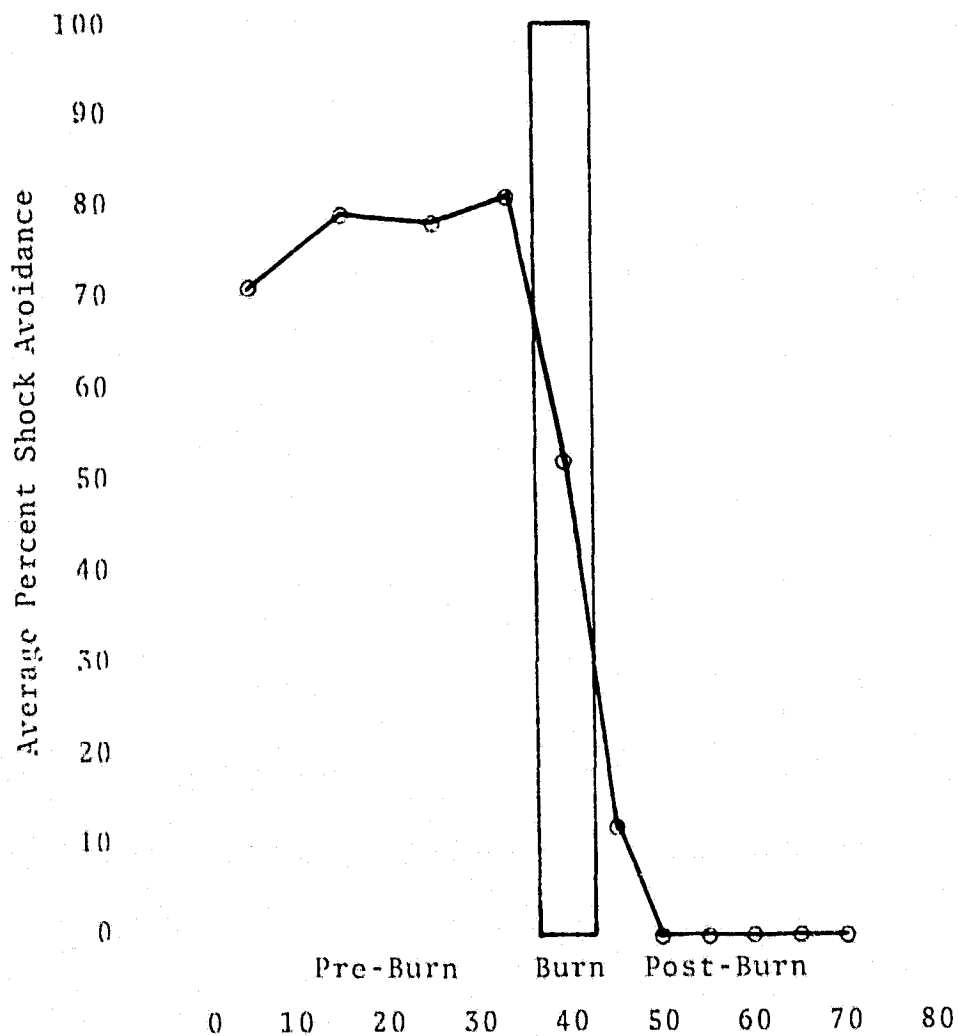


Sample Weight: 18.5 gm.

Maximum Chamber Temperature: 93°F.

Figure 38

Sample Y-7213
Red 20



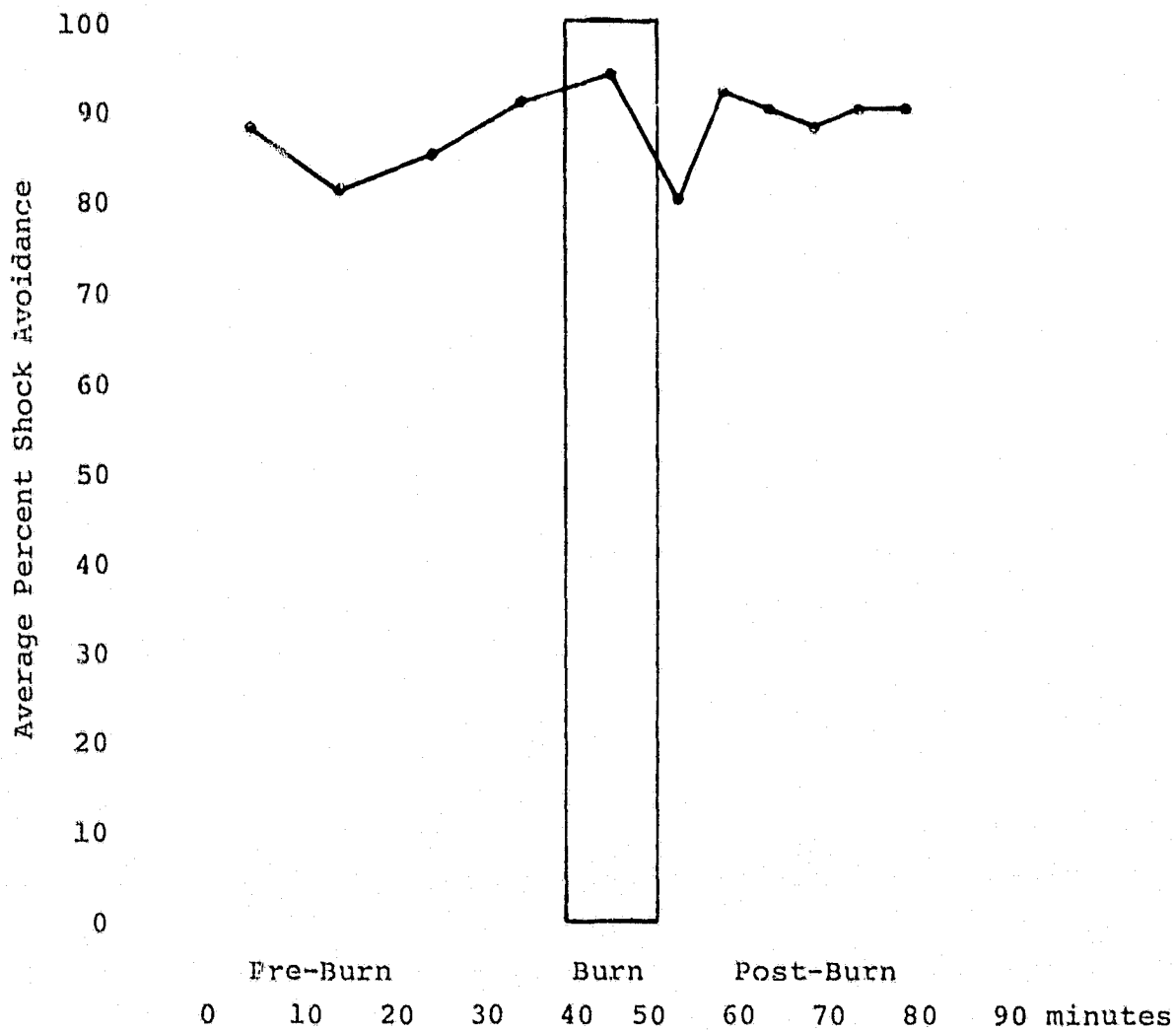
Sample Weight: 18.5 gm.

Maximum Chamber Temperature: 93°F.

Figure 39

Sample Y-7214

Black 3

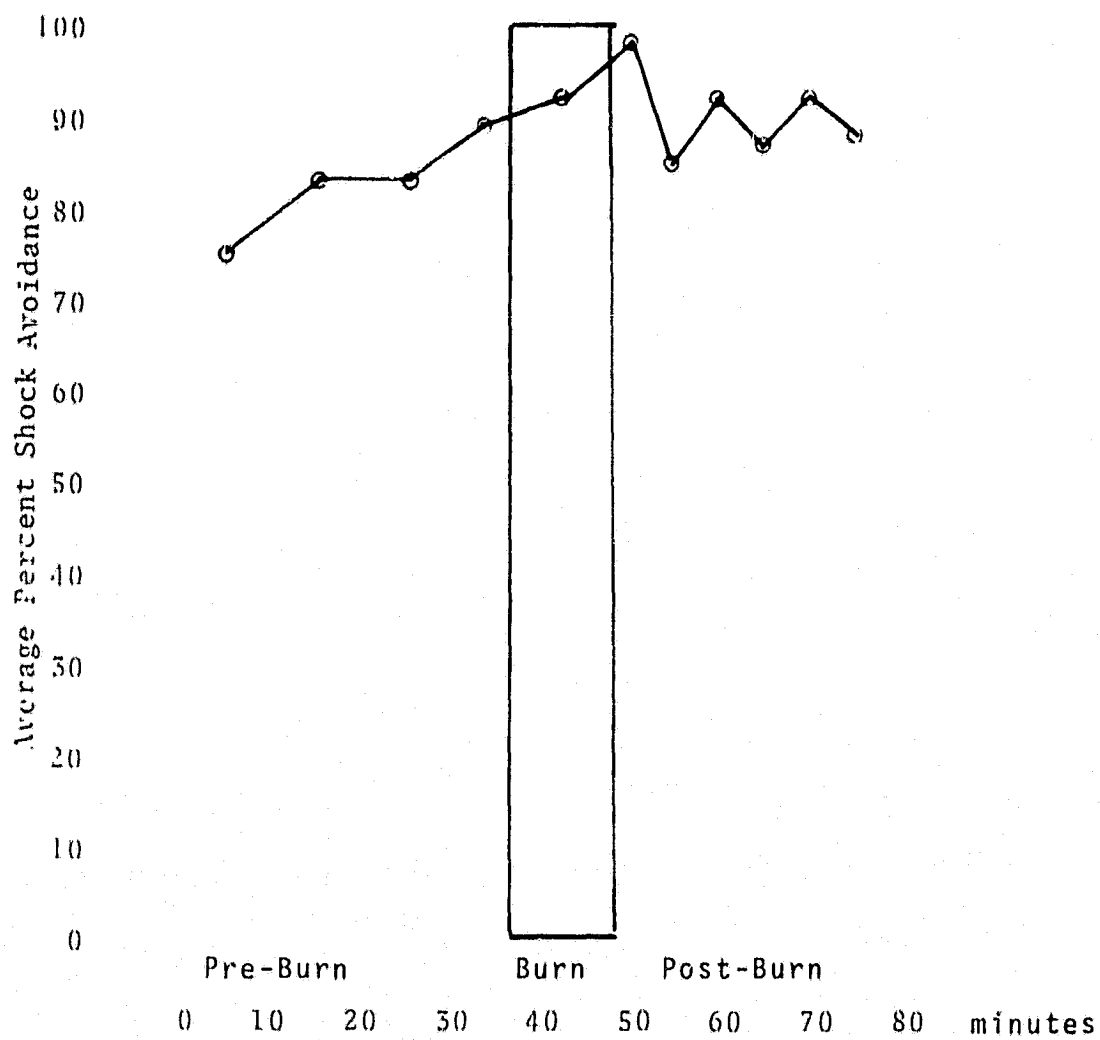


Sample Weight: 2.4 gm.

Maximum Chamber Temperature: 90°F.

Figure 40

Sample Y-7214
Red 1

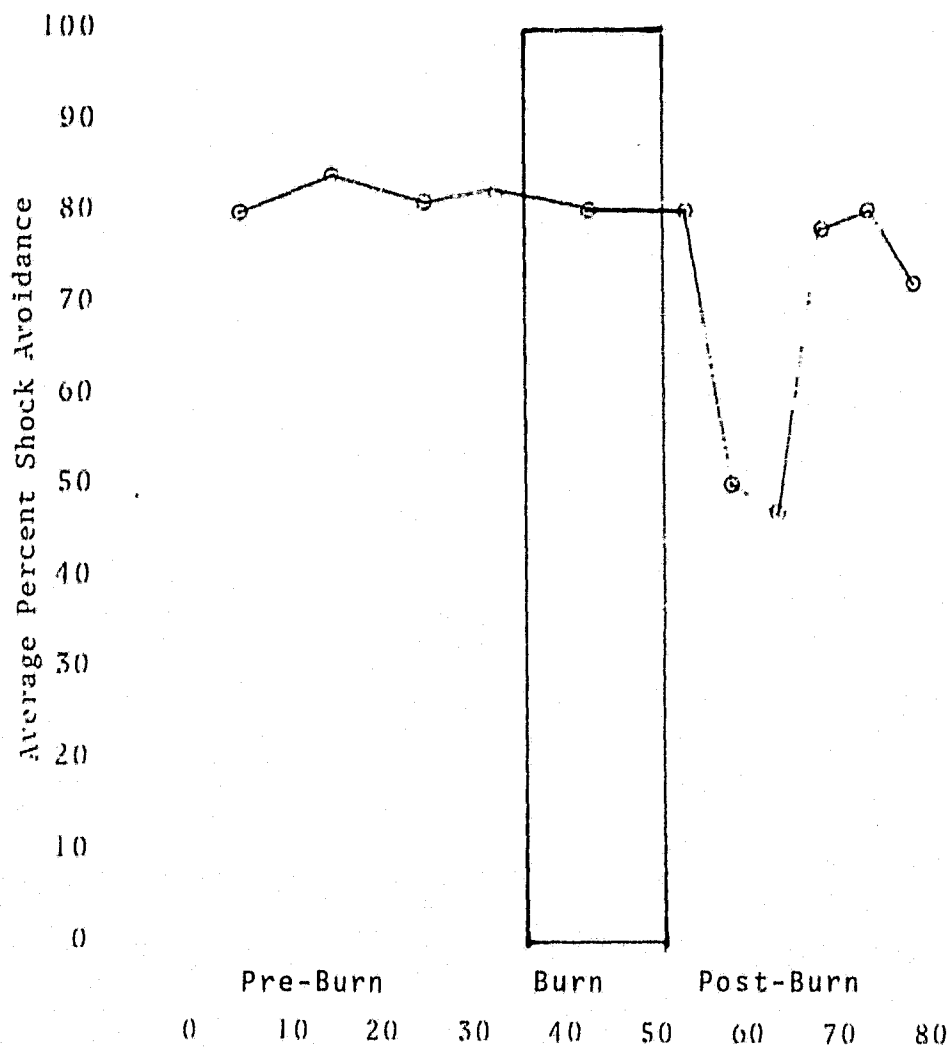


Sample Weight: 2.4 gm.

Maximum Chamber Temperature: 93.5°F.

Figure 41

Sample Y-7214
Red 2



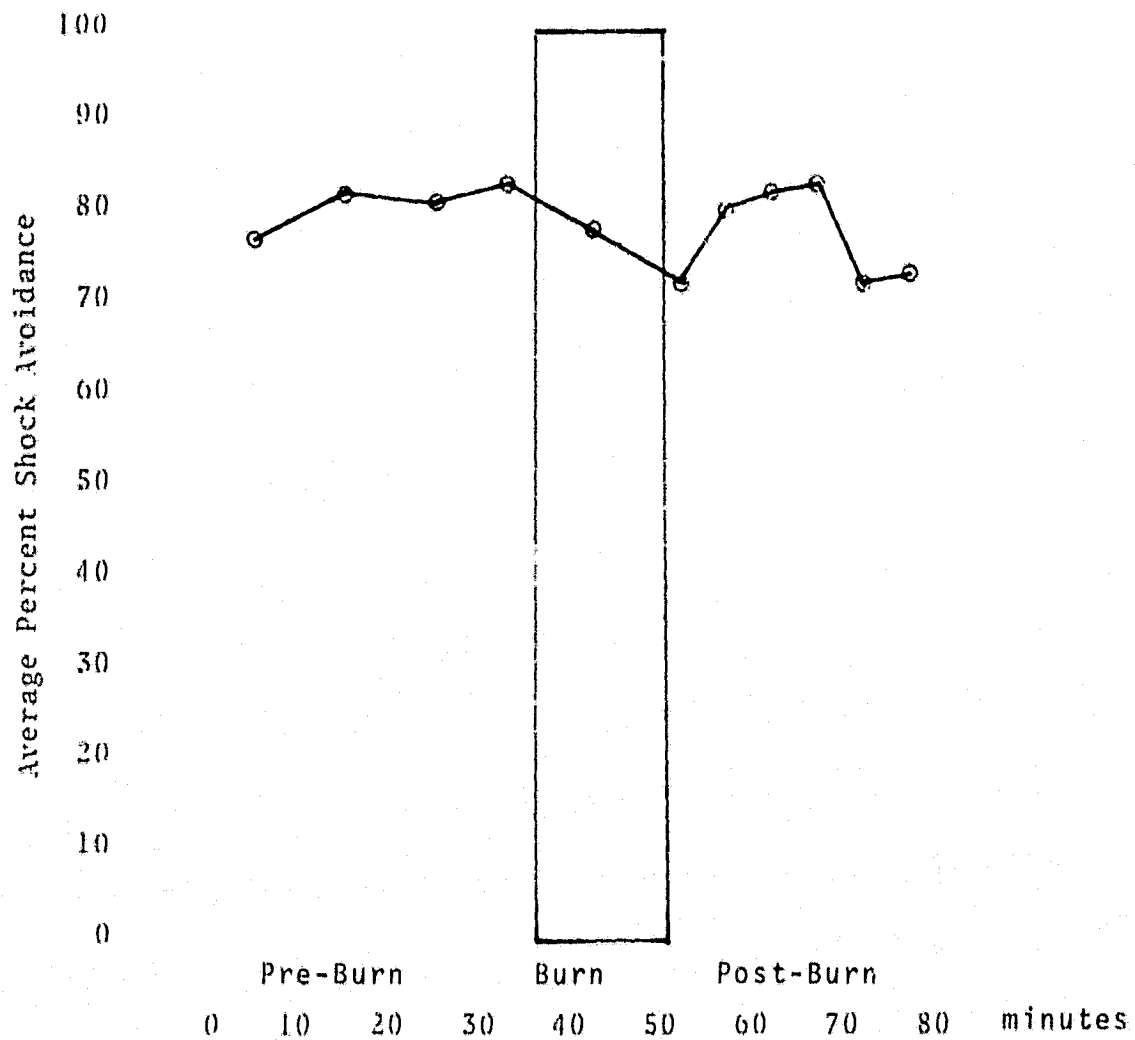
Sample Weight: 2.4 gm.

Maximum Chamber Temperature: 92°F.

Figure 42

Sample Y-7214

Red 14

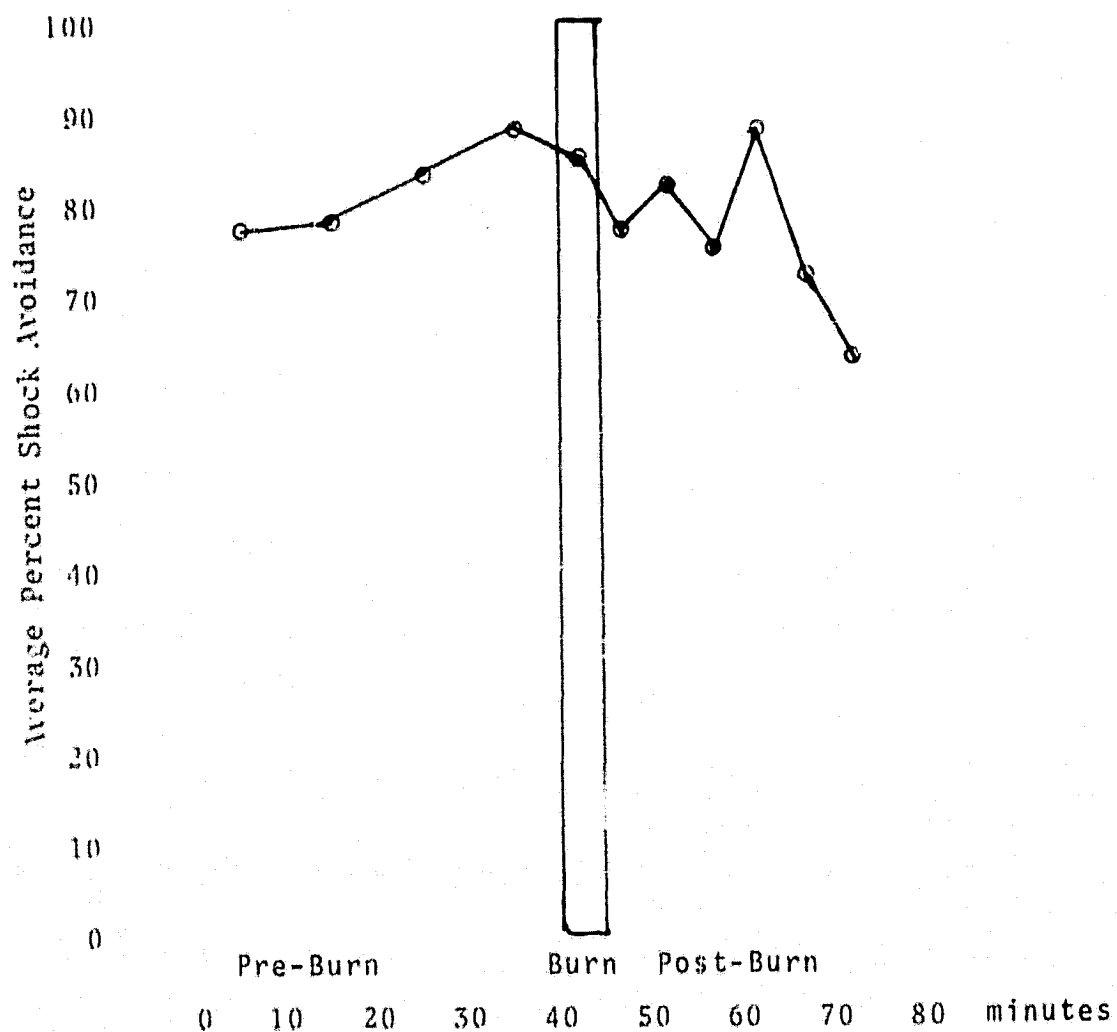


Sample Weight: 2.4 gm.

Maximum Chamber Temperature: 92°F.

Figure 43

Sample Y-7192
Black 9



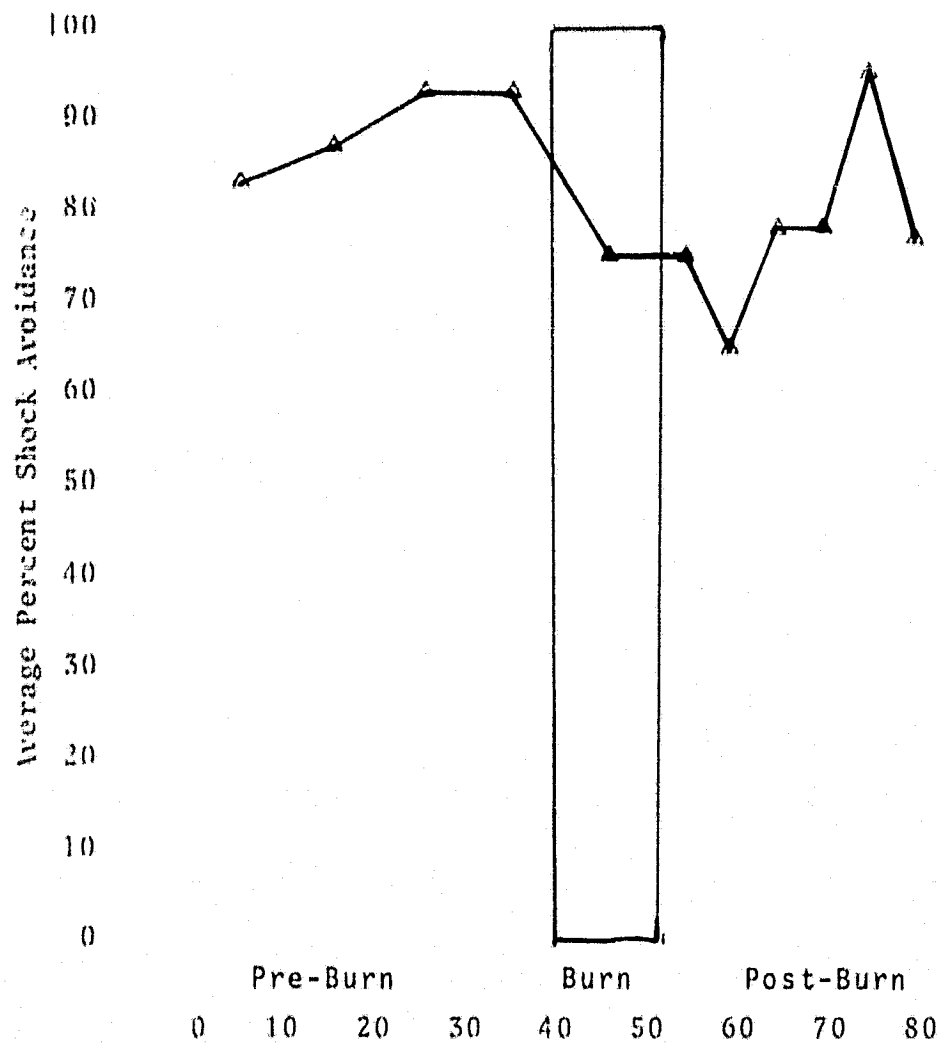
Sample Weight: 1.7 gm.

Maximum Chamber Temperature: 88°F

Figure 44

Sample Y-7192

Red 15

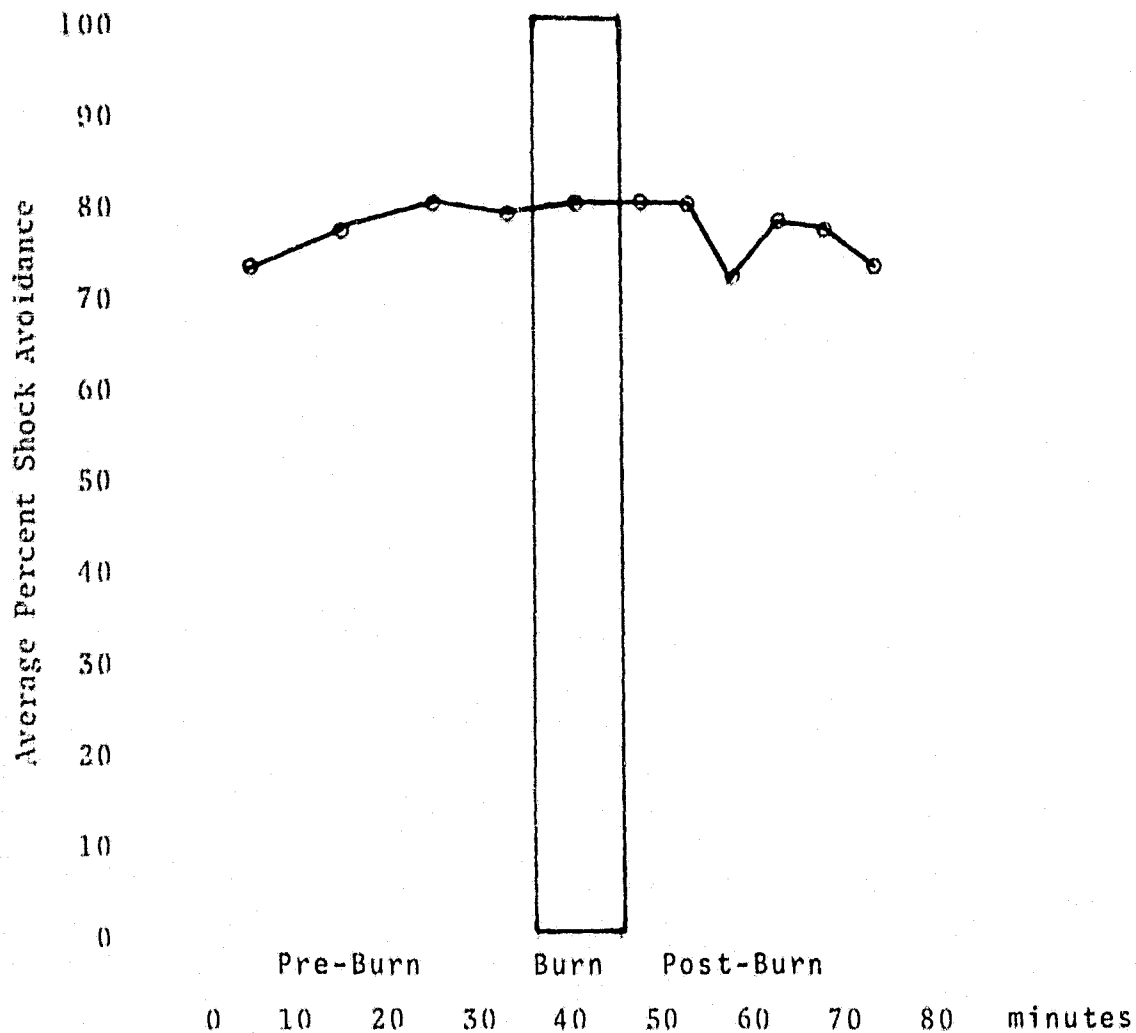


Sample Weight: 1.7 gm.

Maximum Chamber Temperature: 90°F

Figure 45

Sample Y-7192
Red 17

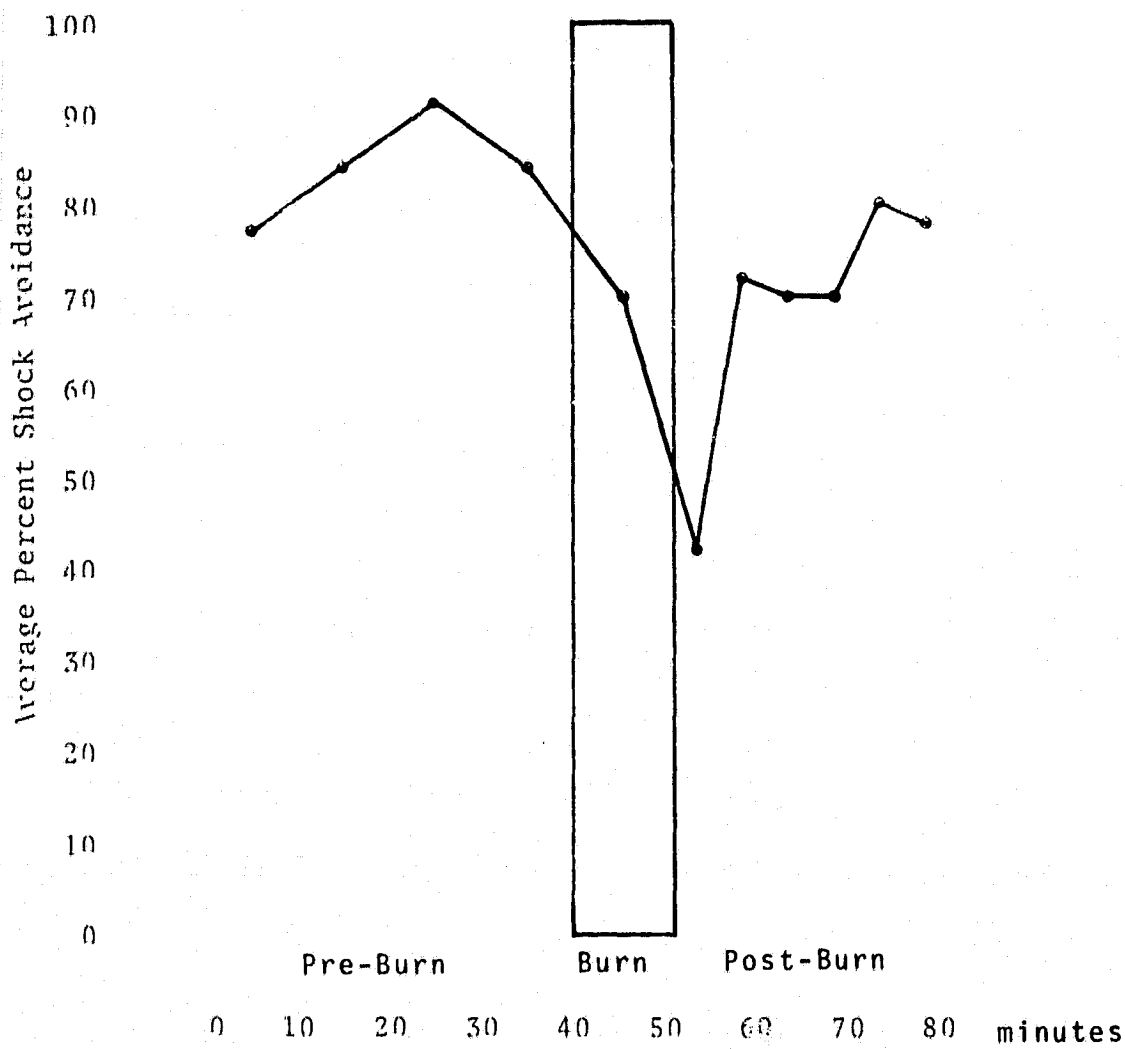


Sample Weight: 1.7 gm.

Maximum Chamber Temperature: 88°F.

Figure 46

Sample Y-7192
Red 19



Sample Weight: 1.7 gm.

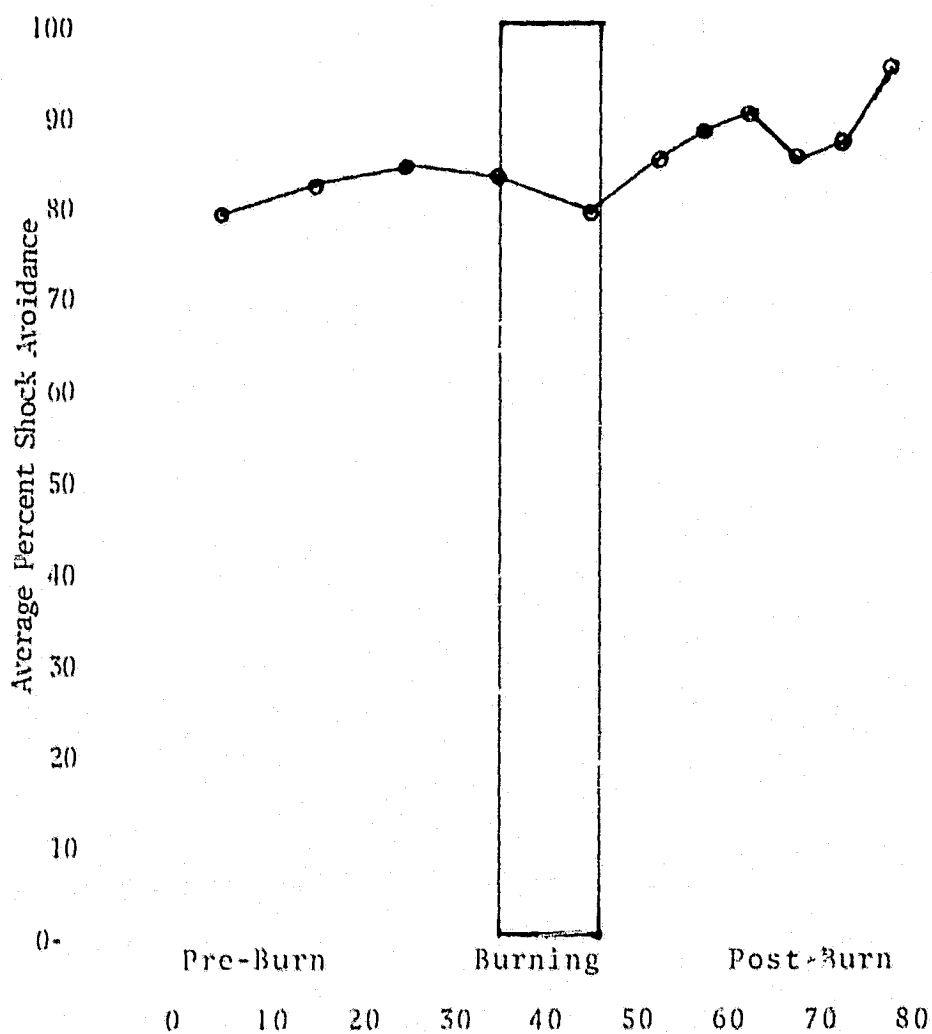
Maximum Chamber Temperature: 86°F.

C-2

Figure 47

Sample Y-7212

Black 7

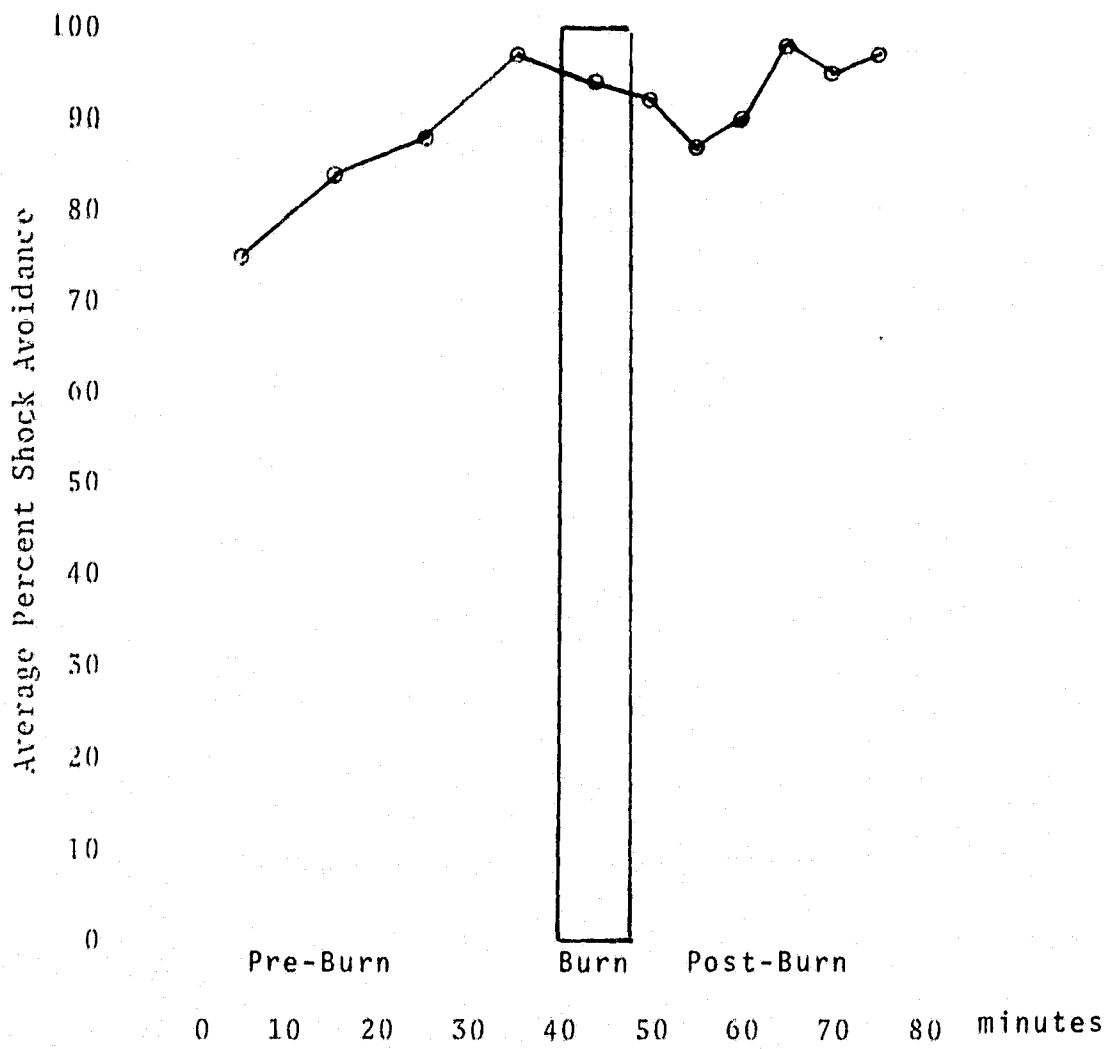


Sample Weight: 1.1 gm.

Maximum Chamber Temperature: 87°F.

Figure 48

Sample Y-7212
Red 4



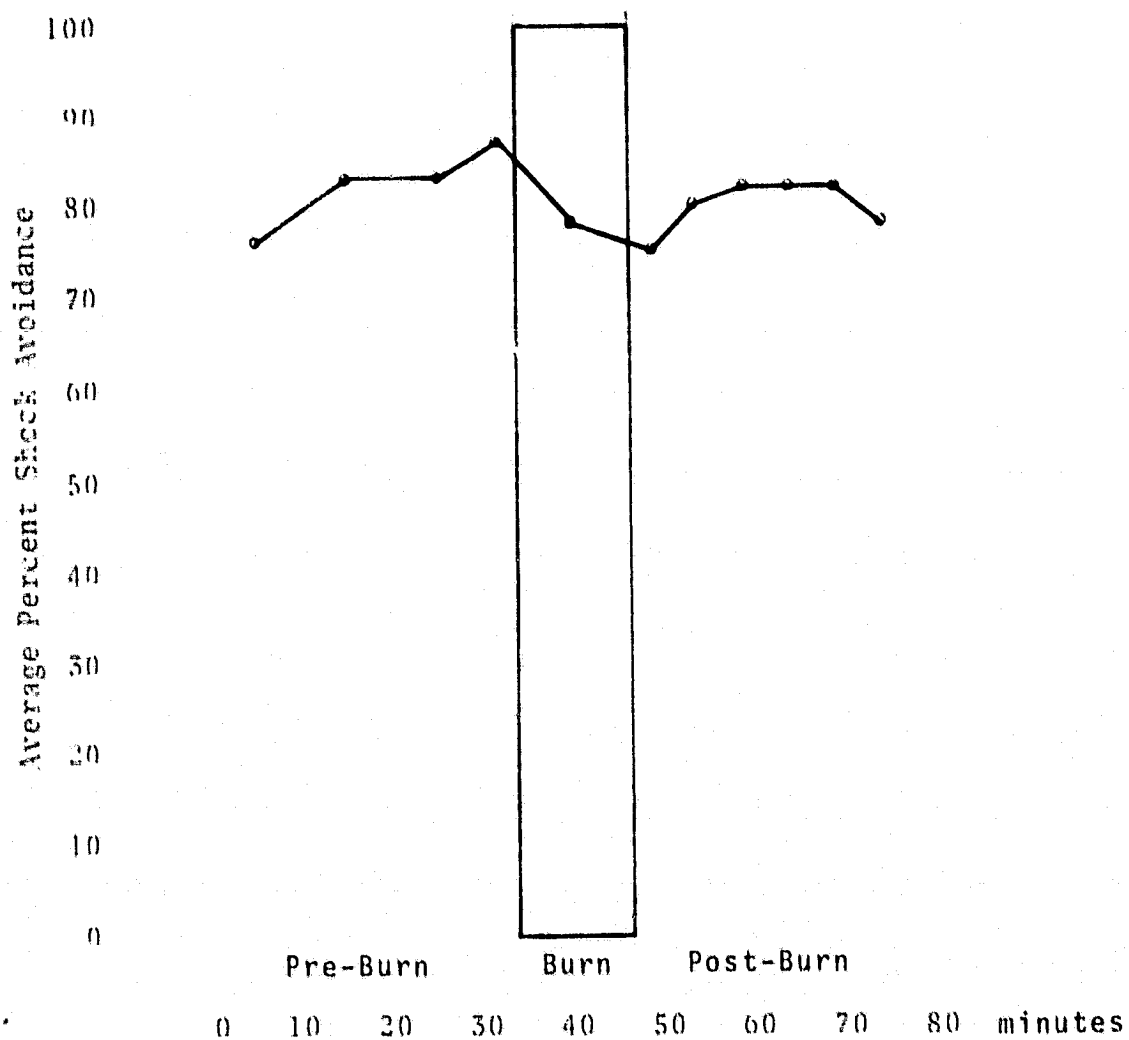
Sample Weight: 1.1 gm.

Maximum Chamber Temperature: 86°F.

Figure 49

Sample Y-7214

Red 25

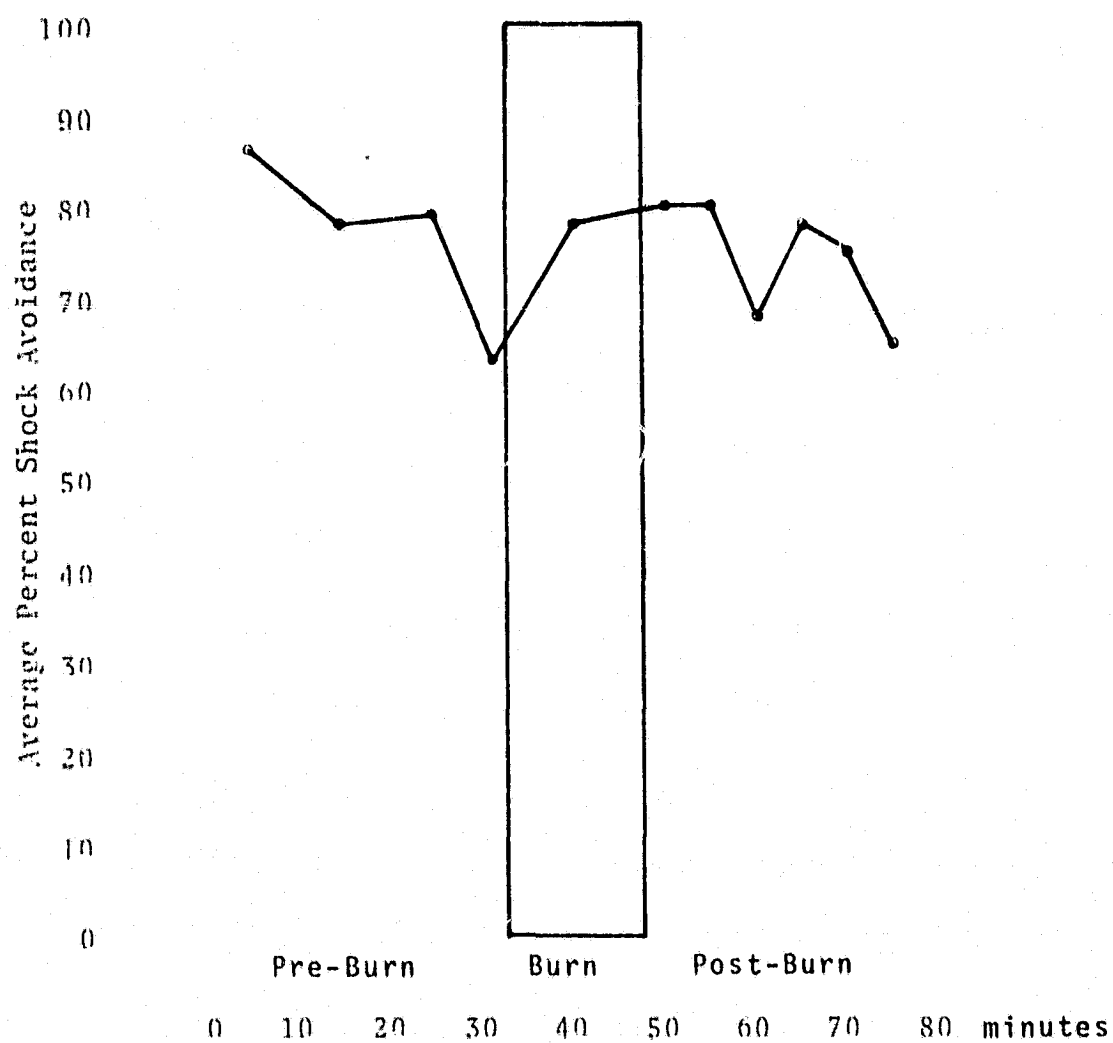


Sample Weight: 3.6 gm.

Maximum Chamber Temperature: 90°F.

Figure 50

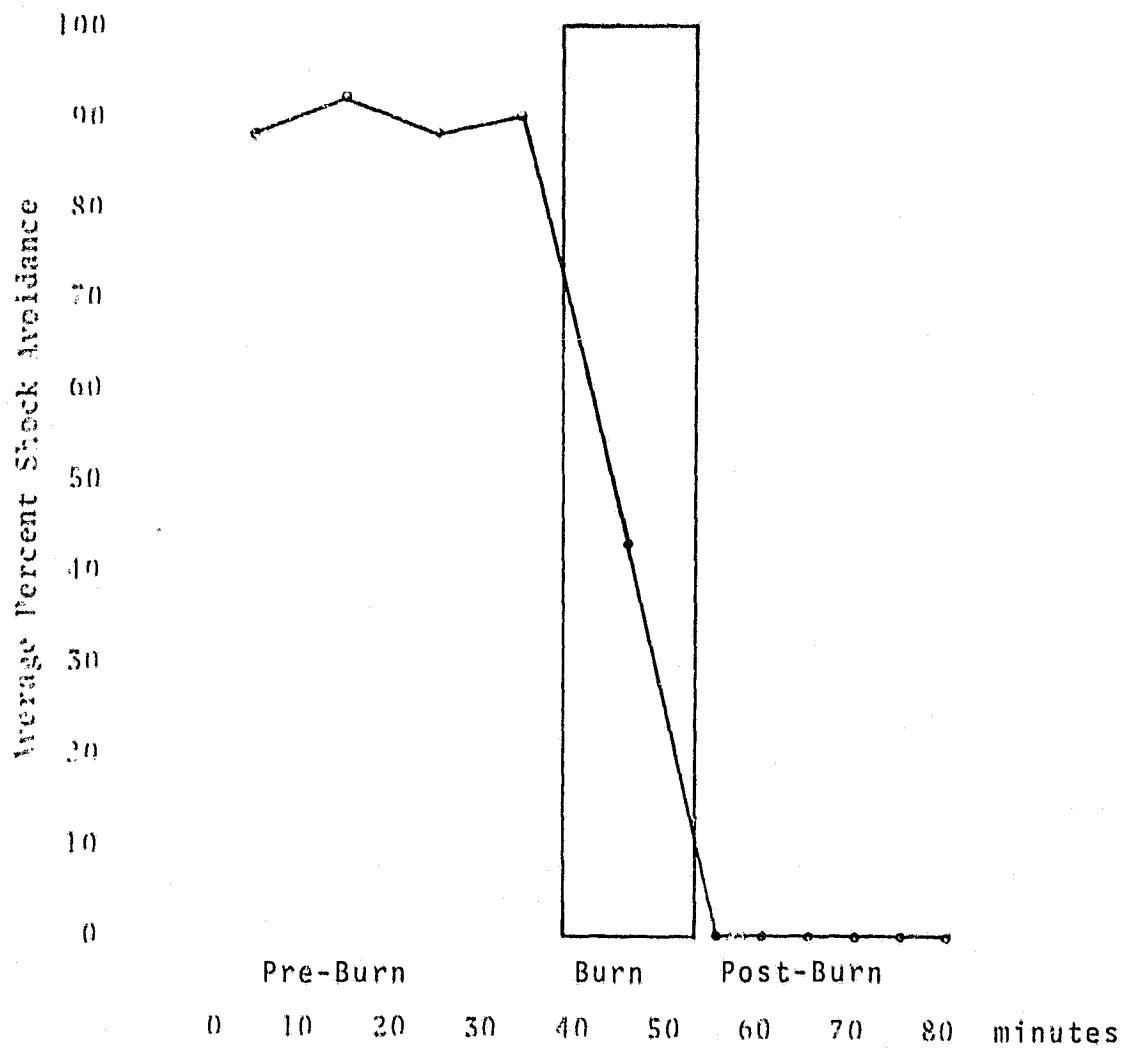
Sample Y-7214
Red 26



Sample Weight: 3.6 gm.
Maximum Chamber Temperature: 92°F.

Figure 51

Sample Y-7214
Red 27



Sample Weight: 3.6 gm.
Maximum Chamber Temperature: 92°F.

EXHIBIT 1

Set 1 - Y-7191

Set 2 - Y-7213

Materials Science Toxicology Laboratories
University of Tennessee Center for the Health Sciences

PT 0.1 10/1 Y- 1191 Data Ref.: 10-11-79 12-16-79

Specimen: _____

Species rat; Group 1; No. 1

Date (Treatment/Sacrifice): _____

Duration: 2; Reason: Glucose 11-11-79

Process Completion Date 10-11-79

AUTOPSY REPORT OR MICROSCOPIC SUMMARY:

☒ BRAIN Congestion acute diffuse mild.

☒ HEART unt

AORTA _____

☒ LUNGS Pneumonitis focal chronic mild. Vasculitis

chronic focal mild

☒ LIVER Congestion acute diffuse mild

GALLBLADDER _____

☒ SPLEEN unt

☒ KIDNEY Congestion medullary acute mild

☒ ADRENAL unt

BLADDER _____

☒ GONADS unt

PANCREAS _____

STOMACH _____

☒ SMALL INTESTINE unt

LARGE INTESTINE _____

TONGUE _____

ESOPHAGUS _____

TRACHEA _____

J. J. J.
Investigator/Technician

29007 88
Date

ORIGINAL PAGE IS
OF POOR QUALITY

32711

Specimen: _____

Species *Agave*; Group *Agave*; No. 1

Date (Treatment/Sacrifice): _____/_____

Duration: 27.5 min.; Reason: Low level of confidence

Process Completion Date _____

AUTOPSY REPORT OR MICROSCOPIC SUMMARY:

16 BRAIN Congestion acute diffuse moderate

XHEART *ind.*

AORTA

LUNGS Atelectasis local moderate. bronchitis
local chronic. mild. Contraction and edema acute

XL TWR Chest in acute softness moderate fever

GALLBLADDER

SPLEEN *unt* *moderate*

• KIDNEY Congestion acute medullary infarct

ADRENAL

BLADDER

✓ GONADS *und*

PANCREAS

STOMACH

SMALL INTESTINE *lin*

LARGE INTESTINE

TONGUE

ESOPHAGUS

TRACHEA

Investigator/Technician

Date _____

27 JUL 82

PT 0. 1737

Y- 7191

page 1 of 2

Deaths

Sample Weight in grams	Group Number	Deaths			MST Path. Number	Histopathology observed which is apparently due to pyrolysate exposure (acute and/or delayed)
		Chamber	14-day Sacrifices	Other (specify day)		
2.048	8, #1	--	4*	--	11710	Vasculitis, chronic diffuse moderate Hemorrhage acute massive Form debris
2.048	8, #2	--	4*	--	11711	Vasculitis, chronic, focal, severe
2.289	12, #1	--	4*	--	11717	Vasculitis, chronic focal mild Pneumonitis focal chronic mild
2.289	12, #2	--	4*	--	11718	WNL Vasculitis chronic focal mild
2.56	9, #1	1*	2	1@1day	11707	Massive Hemorrhage acute
2.56	9, #2	1	2*	1@1day	11712	
2.56	9, #3	1	2	1@1day	NONE	
2.86	11, #1	--	4*	--	11713	Vasculitis, chronic diffuse moderate Pneumonitis chronic focal mild Hemorrhage acute massive
2.86	11, #2	--	4*	--	11714	Pneumonitis focal chronic mild

Additional Comments: X Form debris

PT 0.1737

Y- 7191
page 2 of 2

Sample Weight in grams	Group Number	Deaths			MST Path. Number	Histopathology observed which is apparently due to pyrolysate exposure (acute and/or delayed)
		Chamber	14-day Sacrifices	Other (specify day)		
2.86	20, #1	4*	--	--	11747	Pneumonitis chronic, Fecal Moderate
2.86	20, #2	4*	--	--	11748	Congestion and edema, Fecal Moderate
2.86	24, #1	4*	--	--	11751A	Congestion acute diffuse mild
2.86	24, #2	4*	--	--	11752	Chronic pulmonary disease
3.20	10, #1	4*	--	--	11708	Congestion and edema acute diffuse moderate
3.20	10, #2	4*	--	--	11709	Chronic pulmonary disease
4.0	7, #1	4*	--	--	11705	Congestion and edema acute diffuse moderate - ATelectasis focal moderate
4.0	7, #2	4*	--	--	11706	Chronic pulmonary disease

Additional Comments:

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OF POOR QUALITY

Materials Science Toxicology Laboratories
University of Tennessee Center for the Health Sciences

PT O. 1737 Y-7213 (syms) Data Ref.: MSF # 11852

Specimen: ORGANS

Species CD-1 ; Group 31 ; No. 1

Date (Treatment/Sacrifice): 1/21/80 / 2/4/80

Duration: 2 wks ; Reason: scientific

Process Completion Date _____

AUTOPSY REPORT OR MICROSCOPIC SUMMARY:

✓ BRAIN congestion and edema, acute diffuse m/f

✓ HEART inf

AORTA _____

✓ LUNGS pneumonia, chronic, focal moderate

✓ VASCULATURE chronic, focal m/f

✓ LIVER congestion, acute, diffuse m/f

GALLBLADDER _____

✓ SPLEEN inf

✓ KIDNEY chronic, small, diffuse moderate congestion

✓ ADRENAL inf

✓ BLADDER inf

✓ GONADS inf

✓ PANCREAS inf

STOMACH _____

✓ SMALL INTESTINE inf

LARGE INTESTINE _____

✓ TONGUE inf

✓ ESOPHAGUS inf

✓ TRACHEA NS

J. Turan
Investigator/Technician

26 OCT 80
Date

Materials Science Toxicology Laboratories
University of Tennessee Center for the Health Sciences

PT 0. 1731 Y-7113 48 gms Data Ref.: MST-11/12/4

Specimen: ORGANS

Species ♂ S-D RAT; Group 56; No. 1

Date (Treatment/Sacrifice): 4/7/80 / 4/7/80

Duration: 30 min; Reason: Death inside chamber

Process Completion Date _____

AUTOPSY REPORT OR MICROSCOPIC SUMMARY:

✓ BRAIN Congestion diffuse acute mild.

✓ HEART unt

AORTA _____

✓ LUNGS MASSIVE INSPIRED DEBRIS

✓ LIVER _____

GALLBLADDER _____

✓ SPLEEN unt

✓ KIDNEY Congestion, acute medullary, moderate

✓ ADRENAL unt

✓ BLADDER unt

✓ GONADS unt

✓ PANCREAS unt

STOMACH _____

✓ SMALL INTESTINE unt

LARGE INTESTINE _____

✓ TONGUE unt

✓ ESOPHAGUS unt

✓ TRACHEA unt

J. Turner
Investigator/Technician

29 Oct 80
Date

PT 0. 1737

LD50 not calculated this sample

Y- 7213

Deaths
page 1 of 1

Sample Weight in grams	Group Number	Chamber	14-day Sacrifices	Other (specify day)	MST Path. Number	Histopathology observed which is apparently due to pyrolysate exposure (acute and/or delayed)
5.0	31, #1	-	4*	--	11852	Pneumonitis, Chronic Focal Moderate Chronic Vasculitis Focal mild
5.0	31, #2	--	4*	--	11853	Pneumonitis Chronic Focal mild Pneumonitis Chronic Focal mild
25.0	32, #1	--	4*	--	11854	Pneumonitis Chronic Focal mild Vasculitis Focal Chronic mild
25.0	32, #2	--	4*	--	11855	Not Submitted
45.0	56, #1	4*	--	--	11914	Massive Inspired Debris
45.0	56, #2	4*	--	--	11915	massive inspired Debris

Additional Comments: